THE COMPLEAT DISTILLER

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FOREWORD

Worldwide, there is a growing interest in distillation by those who enjoy making alcoholic beverages, essential oils, botanical extracts and perfumes. We have read books that purport to describe how distillation works, but got it wrong in whole or in part, and some that detail how to build a particular type of still and operate it, but which are based more on myth than sound scientific and engineering knowledge. We came to the conclusion that there was a crying need for a book that gives a detailed account of the entire subject, avoiding old wives' tales and dealing entirely in verifiable facts.

Our book describes the science and the principles behind distillation, the practice of small scale distillation, and several designs for different kinds of stills, all thoroughly tested. We fervently hope that our readers develop a thorough understanding of the process, which can only broaden and deepen their enjoyment of this activity. Following this philosophy, every subject introduced begins with "how does this work?" and progresses to "how can I do this?".

We must stress that this book is **NOT** an incitement to those who live in countries where distillation of alcohol is illegal to rush out and break the law. Home distillation of alcohol is an ancient and honored tradition in many parts of the world, and is completely illegal in others. The wisdom (or lack of wisdom) of the laws prohibiting small scale distillation are not a subject for this book. If you live where this enjoyable activity is prohibited, you are free to read about it, but not to actually do it. If you feel that these laws are misguided, contact your representatives and make the case for changing the laws, do not break them.

Even where distilling alcohol is prohibited, it is legal to distill with water. However, some countries regulate the size of water stills! You must check carefully on your local regulations before engaging in this activity. Using water to steam distill the essential oils and fragrances from herbs, spices and other botanical materials is a fast growing hobby in its own right, and we devote a whole chapter to this subject. These techniques are as interesting and useful to cooks and perfumiers as they are to people who want to make liqueurs.

We have also included a chapter on workshop techniques, tips, and tricks that can be useful in building stills and many other kinds of equipment. This is not like a book that teaches you how to fly -- it's like a book that also tells you how to make your own airplane – and not just the airplane design of our choosing, but one that **you** yourself can learn to design, and which may well fly better!

In this vein, we also describe distillation techniques and designs that we have only recently developed and which have never before been published, and encourage readers to carry the torch further by using their own ingenuity.

One of the authors used an old industrial vacuum cleaner as his first boiler, and later investigated the possibilities of using jam jars to make a glass column. This provoked much mirth and not a little scorn from those who considered themselves 'experts', who considered anything new to be heresy . However, a great deal was learned about the properties of materials and techniques to handle them, knowledge that later proved invaluable. When you read this book, you will see what can be accomplished if you dare to step outside the frame and question preconceived notions of what can be done, and how. All you need is a good understanding of basic principles and a willingness to challenge old, ossified ideas. Home distillation is a fascinating hobby, and one which is fertile ground for those who enjoy exercising their ingenuity. It's challenging, and it's fun!

In Chapter 8, we draw back the curtain on the science behind distillation, a fascinating subject in itself. No myths. No half-baked notions of what we "think" may be going on. Just facts. Facts proven by centuries of hard work and discovery by skilled artisans, scientists and engineers.

Paraphrasing the words of Isaak Walton, the author of "The Compleat Angler", and from whom we have respectfully borrowed for the title of this book:

"Doubt not, therefore, sir, but that distilling is an art,
and an art worth your learning."

CHAPTER 1

FERMENTATION

How alcohol is created

This book is primarily about distillation, the purification and concentration of impure or weak substances. We recognize that the majority of our readers are interested in the purification of ethyl alcohol solutions, and have included this chapter to describe the necessary first steps – the generation of the impure, weak solution to purify! We will not go deeply into the chemistry or biochemistry of the processes, nor will we try to give expert-level advice or directions on any of the traditional processes – very good books already exist on each of these subjects. A list of suggested further reading may be found in Appendix 8.

Synthetic alcohol

One route to an ethanol solution is the production of synthetic ethanol. This is often done industrially, and may or may not be economical in various parts of the world.

Synthetic ethyl alcohol is produced by treating Ethyl Acetate with a strong alkali, or "base" (e.g. lye, Sodium Hydroxide). Ethyl acetate, a common industrial solvent, is an ester, a chemical combination of ethyl alcohol (ethanol) and acetic acid (vinegar). Treating it with a strong base breaks the ester bond, liberating ethyl alcohol and acetic acid. The acid immediately reacts with the base to form the salt sodium acetate. After the pH of the solution is adjusted to near 7 (neutral), the pure alcohol may be distilled from this mixture.

This procedure is NOT recommended unless you have experience handling strong chemicals, and they can be obtained cheaply in your locale. It is really included for completeness – if it wasn't, someone would write us and ask “Why didn’t you mention the ethyl acetate method?”

If the term “pH” is unfamiliar, don’t worry! The pH of a solution is a measure of how acidic or how alkaline it is, with 0 being the most acidic, 7 being neutral (neither acidic nor alkaline) and 14 the most alkaline. The scale is logarithmic, which means that each number on the scale is TEN TIMES the previous one. Thus, pH 5 is ten times as acidic as pH 6, and pH 11 is ten times as alkaline as pH 10. For a few real-world examples, Lemon juice is about pH 2.5 (very acidic), Human skin is about pH 5.5 (very mildly acidic), and liquid soap (the clear kind found in public bathrooms) is about pH 10. For a more complete explanation, please see Appendix 1.

Fermentation

Basic biochemistry

All living things require energy to continue living, and many different ways of obtaining that energy are found in nature. Plants convert the energy in sunlight into chemical energy their cells can use and store. Sugars, starches and fats are the most common energy storage products. Animals, fungi, and most bacteria break down the chemicals that are stored by plants to extract the energy they contain.

There are two fundamental methods of extracting this energy – with and without the assistance of oxygen. Energy production assisted by oxygen is six times more efficient than without it, so cells preferentially use oxidative metabolism whenever possible.

Fermentation is a method microorganisms use for extracting energy from food when oxygen is not available. In fermentation, complex molecules (like sugars) are partially broken down into simpler molecules, and the energy stored in the chemical bonds is released and captured by the cell.

There are many, many different kinds of fermentation, and most of them result in the production of organic acids (acetic acid, lactic acid, butyric acid, etc.). These fermentations are used to produce familiar food products such as sauerkraut, yogurt, many cheeses and black tea.

Some organisms, including many species of yeast, are capable of fermenting to produce an alcohol and carbon dioxide gas. Exploitation of these organisms is one of the oldest of human industries.

No natural organism does just one thing; as yeast grow and produce alcohol, they also produce acids via other types of fermentation. The enzymes that manage the reactions within the cell are also not perfect, and a percentage of the reactions in the cell produce a different result than expected. These mistakes become much more common when temperatures or chemical concentrations are high.

Enzymes are very large molecules with specially-shaped cavities in them. These cavities are just the right size and shape for a particular molecule (or group of molecules) to fit into, and when the molecules sit in the cavity just right, a chemical reaction happens. This process is driven by the energy present in all material – the constant collisions of molecules against one another, millions of times a second. The more concentrated a substance is, the more chances for it to collide with another molecule, whether in the correct position or not. The higher the temperature, the more forceful the collisions, which makes it more likely that an incorrect position (or an incorrect molecule) might lodge in the cavity, leading to an undesired reaction.

Another key point about biological (enzymatic) reactions is that they can run in both directions! An enzyme that makes a compound can also unmake it, and the end product of the reaction fits in the pocket in the side of the enzyme just as nicely as the starting compound. The reverse reaction is usually slower than the forward one though, which is why we can obtain an end product!

This means that these reactions slow down as their end products build up and the starting products are consumed – it becomes more likely that the enzyme that turns acetaldehyde into alcohol will encounter an alcohol molecule than one of acetaldehyde. In the same way, the acids produced by the yeast also build up, lowering the pH of the solution, which makes metabolism and growth more difficult. (This is the reason many preserved foods, like pickles and sauerkraut are acidic - bacteria have a very hard time at low pH).

Congeners (fusel alcohols)

("Fusel": German for "rotgut" or "hooch")

The molecules made by mistake tend to be longer chain alcohols, acids and esters, all of which add flavors (usually unpleasant) to the product, and many of which are toxic. These are called “congeners,” which means “with from the birth” – they were produced at the same time and by the same processes as the alcohol we desire. The composition of these congeners, collectively known as "fusel oils", or more correctly fusel **alcohols**, varies with the carbohydrate source and yeast variety, but is mostly isopentanol and 2-methyl-1-butanol, with about 20% isobutanol, 3-5% n-propanol, and small amounts of other alcohols, esters and aldehydes. Carefully selected strains of yeast and carefully managed temperatures and concentrations result in cleaner, more pleasant fermentations. Since the rate of chemical reactions generally doubles for every 10º C (18º F) temperature rise, high quality fermentations conducted at low temperature take more time. This compromise of time vs. quality is present everywhere in the production of alcohol-containing beverages. Managing the conditions of fermentation is the primary method of producing specific flavor profiles in fermented beverages.

Many different sources of sugar may be fermented into alcohol, and many others either will not ferment, or cannot be fermented by particular yeasts. Differences in the type of the sugars available, the species of yeast available and the quality of the local water have led to a huge variety of different types of alcohol-containing beverages around the world. The next section of this chapter discusses a few of the more common types, and how to prepare them for distillation.

Properties common to all fermentations

All successful alcohol fermentations share many properties in common, which are discussed here. In the sections on fermentations for specific purposes, we discuss the differences in procedure that produce the special flavors and characteristics of individual beverage types.

No matter what kind of product you are making, some things are always the same, and these are:

- All ethanol fermentations require a source of sugar to ferment, and have limits to how concentrated or weak that sugar can be.
- All ethanol fermentations will do best with rigorous cleanliness and sanitation.
- All ethanol fermentations require oxygen in their early phases, to promote the growth of yeast. (However, air MUST be kept out after the first day or two, or no alcohol will be made!)
- All ethanol fermentations produce heat. This is usually not a problem with a small batch, but temperature management can be a critical issue with larger volumes.
- All ethanol fermentations produce carbon dioxide gas, which is poisonous. Again, with small batches, this is not an issue, but with larger batches ventilation can be necessary. Every kilogram (2.2 pounds) of sugar will produce about 250 liters (8 cubic feet) of carbon dioxide over the span of the fermentation.

Various types of fermentation

Beers and Whiskeys

Beers are fermented products made from cereal grains. Grains store starch (a long-chain molecule made by stringing together large numbers of sugar molecules) as an energy supply. Starch is not fermentable but, when it is broken down, many of the resulting sugars are. Allowing the grain to sprout (called “malting”) creates enzymes that break down the starch into sugars and shorter-chained sugar polymers (dextrins).
When this mixture (called a “mash” while it contains the grains, or a “wort” if the liquid portion is separated from the grains) is fermented, the product is known as a beer. When a beer is distilled, the product is known as whiskey. Beer intended for whiskey making is often fermented as a mash rather than a wort, because the distillation process will separate out the starch, proteins and other contents that are not desirable in a beer intended for drinking.

Extracts of malted grain are highly nutritious, for yeast as well as for people! Malt extract is in fact almost the ideal environment for yeast - lots of sugars, lots of vitamins and minerals, and buffers (mixtures of compounds which keep the pH within a comfortable range during the fermentation). Consequently, beer fermentation may be quite rapid. However, being slightly acidic and well-buffered at pH 4.5 to 5.5, malt extract is also an ideal food for other undesirable microorganisms, and beers can be easily spoiled by insufficient sanitation.

Wort is almost always boiled vigorously as the first step to making a beer, to coagulate the excess proteins that would cloud the beer and to kill all the bacteria present in it. After it is cooled, a large quantity of the desired yeast is added, and fermentation proceeds for a few days to a few weeks, depending upon temperature. For the homebrewer, the most common fermentation vessel is a 20 liter (5 US gallon) glass carboy. This is a convenient size, is easy to clean and sanitize, and can easily be fitted with an airlock to keep oxygen out while allowing carbon dioxide to escape.

Meads

Meads are fermented from honey, The most concentrated natural source of sugar. Honey is a mixture of natural sugars, and does not ferment by itself because the concentration literally pulls water out of bacterial or yeast cells (a process called "dessication"), preventing them from growing. This tendency of concentrated sugar solutions to pull water out of other things is called “osmotic potential”, and can have a profound impact on the survival and fermentation rate of yeast. The basic mead technique is to dilute honey to the proper sugar content, heat or boil it gently to kill wild bacteria and yeast, and then inoculate with a large quantity of yeast.

Since honey is essentially pure sugar, it is also very low in the vitamins and minerals so abundant in malt extract. It also has no buffering capacity, so the acids produced by the yeast during fermentation will rapidly reduce the pH and slow down the fermentation. Consequently, mead has a reputation of being very difficult and time consuming to make, and is not attempted by many people.

Home mead-makers generally ferment in glass containers, ranging in size from 4 liter (one US gallon) jugs through 20 liter carboys. Glass is especially recommended because it's impervious to oxygen. A plastic container would allow significant amounts of oxygen to diffuse through the walls during the very long, quiet fermentation traditional to mead.

Recently, techniques have been developed to properly feed and manage the pH of yeast growing in diluted honey or sugar that make mead making almost as rapid and reliable as beer brewing. These will be discussed in the section on fermenting techniques.

Wines and Brandies

Wines are the fermented juices of fruit, and when wines are distilled the final product is called brandy. Some fruit wines are commonly called by alternate names – for example, cider for fermented apple juice and perry for fermented pears. Most fruit juices will ferment by themselves if left out in the open air (witness an over-ripe plum), but the results are often not pleasant because many different kinds of organisms are present. Many fruits contain considerable quantities of acid (malic acid in apples, citric acid in citrus fruits, etc.) which not only give them their “tang”, but also inhibit bacterial and yeast growth.

By far the most common wine is made from grapes. Grape juice is quite acidic, and falls between malt extract and honey in nutrient levels. Mature grapes usually have a coating of wild yeast on the outside of the skin, living on the sugar that “leaks” out of the grape. This means that simply crushing the grapes to release the juice will result in the production of a wine – but a wine that will often be sour and unpleasant to drink. These high levels of acidity allow one to make wine with lower sanitation levels than are required for beer and mead, but as a general rule better sanitation will always result in a better wine. Be sure to wash your feet very carefully before using traditional methods!

The standard method of producing wine today is to treat the juice with a product that kills the wild yeast, then to heavily inoculate the juice with a carefully selected yeast strain. Wine fermentation takes significantly longer than beer, but less time than mead. Most home wine makers perform the initial phase of fermentation in a plastic food-grade garbage container, then transfer the partially fermented juice (called “must”) to glass carboys or wooden barrels for the remainder of fermentation.

Root vegetables and Vodka

Any starchy or sugary root, tuber or stem can be converted into fermentable sugars, and all of them are used for this purpose somewhere on the globe. Reading through old manuals on alcohol production will give you details of many processes based on potatoes, beets, turnips, and just about any other starchy plant you can imagine. The basic process is to convert the starch to sugar using enzymes (from malted grain, fungi, or industrial sources), fermenting the sugars to ethanol, then purifying the alcohol through distillation and filtering. Alcohol produced in this fashion is usually used for industrial purposes or is purified again and sold as Vodka (defined in some countries as unflavored neutral ethyl alcohol), because the starting material does not contain useful or desirable flavors that are wanted in the end product.

Sugar-based washes and Rum

Sugarcane is clearly a good source of fermentable sugar. When cane is crushed, the sugar-rich juices are directly fermentable. However, sugarcane juice is not nearly as acidic as fruit juices, so the fermentation is easily contaminated by wild yeast and bacteria, which can have a significant impact on the flavor of the fermented beverage. Distilling fermented sugarcane juice yields aguaardente (strong water), known best as Cachaça.

Sugar is made from sugarcane by boiling the juice down and removing the remaining solids. The thick liquid produced by boiling sugarcane juice is molasses, and distilled beverages produced from fermented molasses are known as rum. Cachaça and rum are fermented and distilled in an amazing variety of equipment all around the world. There is no single, traditional method to quote – and almost any that you can imagine is in use somewhere in the world.

Both sugarcane juice and molasses contain a wide array of nutrients, and need only to be inoculated with yeast to begin active fermentation. Purified sugar, on the other hand, has had the great majority of its nutrients removed and will not support a vigorous fermentation unless nutrients are added, or a huge over abundance of yeast is used.

The Home Distiller's dilemma

Given this fact, you might ask why would anyone want to ferment pure sugar, and the answer is one of the oddities of economics. Due to the nature of distribution channels, purified table sugar is often the least expensive source of fermentable material available to the small-scale experimenter or hobbyist. A home distiller intent upon making pure ethyl alcohol could use any of the above starting materials, but the cheapest, easiest and most available is pure white granulated table sugar. Where this is not the case, the cheapest and most readily available sugar source is sure to be used.

Home distillers wishing to make specific distilled spirits, like schnapps, brandy, whiskey, etc. will need to utilize the proper starting materials. However, be warned that the final taste of many of these products is produced at least as much by the aging process of the spirits as it is by the starting material. These aging conditions are often not possible to duplicate at home, so caution and patience are necessary when trying to duplicate a commercial product.

The art of fermentation

The Basics

Fermentation requires several things – sugar within certain concentration ranges, healthy live yeast, proper temperatures and nutrients to allow the yeast cells to increase in number and remain healthy throughout the fermentation process. A good, controlled fermentation with complete conversion of the sugars to alcohol but producing no bad or "off" flavors, requires a few more:

- Good sanitation and cleanliness
- Selection of the best yeast variety
- Using a large quantity of that yeast
- Proper fermenter geometry
- Fairly precise control and management of temperature, pH and oxygen levels

Don't despair, though – in normal hobbyist quantities, these are all fairly easy to arrange.

In many countries, home beer, mead and wine making are well established hobbies, and many stores provide a wide variety of materiel and equipment to carry out fermentation. Let's take a look at this equipment and consider how it may be used for our purposes. Later on, we'll examine the possibilities for higher volume and more intensive fermentation.

Fig. 1-1

The single most common fermentation vessel in use is a glass carboy sized to contain 12 to 25 liters (3 to 6 US gallons) of fluid. In the USA, the five gallon carboy is the overwhelming favorite, because it's cheap, conveniently sized, and readily available. The advantages of the glass carboy are: it's clear (which means progress can be followed without opening it), is easy to sanitize, and has a small opening in which it's easy to fit an airlock for oxygen control. The most obvious disadvantage is that it is breakable – both by physical impact and by exposure to extremes of temperature (don't sterilize with boiling water!) The carboy also allows a single-stage fermentation to be performed when there are no solids that need to be removed part way through the process (grains, grape skins, etc).

Fig. 1-2

The second most common fermentation vessel is a plastic bucket or trash container made out of food grade polyethylene or polypropylene. These range in size from 20 to 80 liters (5 to 20 US gallons), and are more commonly used in wine making than in beer or mead. The advantages of the plastic containers include the facts that they are open – materials can be easily added to or taken out of them – and that they are unbreakable. The disadvantages are: they are harder to sanitize than glass, especially if the plastic is scratched, oxygen diffusion through the plastic may limit their safe use to short fermentations, and they can also absorb bad odors or flavors and later impart these to the fermenting ambrosia.

Both plastic buckets and trash containers have reasonable geometry for unstirred fermentation – one and a half to two times taller than they are wide. This allows the fermenting fluid to circulate due to the action of the CO_2 produced. Shallow, wide fermenters can be successfully used, but generally need some sort of mechanical stirring.

A fermentation started in an open container (even if fitted with a loose lid) should be finished in a second, closed container – a carboy, barrel, or stainless steel tank, to allow control of oxygen levels.

Accessory equipment needed to do a good job of managing fermentation includes accurate thermometers, hydrometers, pH test paper, airlocks, hoses for siphoning liquid from vessel to vessel, funnels, stoppers and cleaning supplies. These are the basic items and can be purchased very cheaply. For the enthusiast, more sophisticated equipment is available from hobbyist shops and specialized manufacturers. Countless resources are available, including many good books, catalogs, websites and magazines that detail how to use all of this gear to maximum advantage.

Many of the precise procedures used will depend upon the material being fermented, and the flavors desired in the final product. In this book, we're mainly concerned with the basics of fermentation and don't pretend to offer advice on how to produce fine beer, wine or mead. If your interest is sparked by that absorbing pastime, then you'll find dozens of excellent books covering these subjects in depth.

Single stage fermentations

Single stage fermentation entirely in glass is possible for beers made from malt extract or separated wort, wines made from purified or filtered fruit juices, honey-mead, molasses, and other sugar-based washes. The general technique is to sanitize the container and airlock, rinse thoroughly with clean water, introduce the fluid to be fermented together with the yeast, aerate, attach an airlock device and wait for initial signs of fermentation to begin - usually 12 to 24 hours. The complete fermentation can take from as little as 4 or 5 days for beers, and up to 6 months or longer for mead.

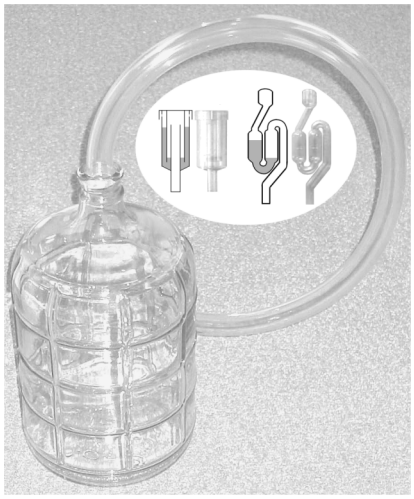


Fig. 1-3

Fluids which contain a lot of protein (especially grain-based wort) tend to foam a lot in the early stages of fermentation, so a special kind of airlock, called a blow-off tube is used until the fermentation settles down. These fermentations should be performed in a container larger than the final volume being fermented, or a significant quantity of product can be lost during the initial, vigorous fermentation. Alternatively, two-stage fermentation can be used for these types of fluids.

Two stage fermentations

Where solids (peels, grains, twigs and stems, other ingredients, etc.) or proteins are present in large quantities, a two-stage fermentation can be very useful, and is traditional for wine production.

An initial “primary” fermentation is carried out, usually in an open fermenter. An open fermenter is usually fitted with a lid to keep the precious fluid clean, but since this is not an effective airlock it can be regarded as being open to the atmosphere. After the initial foaming has died down, or the color and flavor are extracted from the peels or other ingredients, the still-fermenting liquid is carefully siphoned off into a “secondary” fermentation vessel, often a glass carboy. This procedure is called “racking.” The remainder of the fermentation takes place away from the air in the protected environment of a carboy, barrel or fermentation tank carefully sealed with an airlock (which may be a firmly secured cork for the last stages of fermentation of sparkling wines).

Special fermentation techniques

Many industrial techniques have been developed to allow faster fermentation and stronger products, producing greater quantities of alcohol in less time from cheaper and lower quality substrates. Some of these techniques can be very useful to the production of beverage alcohol, either for direct consumption or for distillation.

Strain selection

The biggest contributors to the final flavor of the fermented or distilled product are the strain of yeast selected and how that strain is treated during the fermentation.

Different strains of yeast make different congeners in widely different quantities. They also have very different responses to temperature, presence of specific nutrients, oxygen levels, etc. There is a considerable amount of information available about yeast strains and varieties, in books and magazines as well as from the yeast suppliers. It's a very good idea to get as much of this information as possible and to use it carefully when planning a production run for a particular product. Some sources of this information are listed in Appendix 8.

Home-brewing clubs frequently experiment with this phenomenon. In these experiments, a large batch of wort is made and prepared for fermentation, and separated into several carboys, each of which is inoculated with a different yeast strain. After completion, a tasting is held to compare the results. The differences can be staggering. Similarly, a single strain of yeast might be utilized at a variety of temperatures, with results ranging from a very crisp, “lager-like” beer produced at low temperatures to an extremely fruity, “estery” brew at higher ones.

Different strains of yeast also have very different temperature and alcohol tolerance levels. All yeast will die at some level of alcohol concentration, and this tolerance level drops as the temperature rises. Baking yeast, for example, dies off quite quickly when alcohol levels reach about 6–7 %, rendering it useless for making beverage alcohol. Most strains of Beer Yeast can tolerate 8-10% alcohol, though some can take more. Wine Yeast strains generally can ferment up to 14-15%, and a few can go as high as 20%. Adding large quantities of the wrong yeast will only make it reach its tolerance point faster, it will not result in a higher final alcohol concentration. There are hundreds of different strains of wine, beer and specialty yeast available from a large number of suppliers. Matching the strain to the desired product will greatly improve the success of your fermentations.

Nutrient, oxygen and yeast management

Dr. Clayton Cone, of Lallemand (a major supplier of yeast for all varieties of fermentation) has spent years studying “difficult” fermentations (honey, corn syrup, rice syrup, cane sugar, ultra-filtered fruit juices, etc), and determined these all shared a number of common problems:

1. Very little available nutrients, B vitamins and particulate matter for the yeast.

   Although yeast are largely self-sufficient, they need some vitamins (especially B vitamins), mineral nutrients (especially nitrogen, phosphorus and zinc) and particles for them to stick to, (which helps to keep them suspended in the fermenting fluid.)

   Commercial yeast nutrient powders supply the nutrients and particles that yeast need to thrive. Examples are Lallemand’s Fermaid K® and Wyeast Lab's yeast nutrient. These contain vitamins, minerals, amino acids, free amino nitrogen compounds, zinc, phosphates, dead yeast shells and other trace elements. Optimum yeast nutrition will also require the addition of diammonium phosphate (DAP) during the fermentation.

2. Little or no buffering material to stop the pH from dropping rapidly during the first few hours.

   Yeast produce significant amounts of acid as they ferment, which can lower the fluid pH. Natural juices and worts contain buffering materials, which limit how low the pH can go. Ferments without buffering capacity can drop as low as 2.7, which can stress the yeast so badly it never recovers. A pH below 4.5 is good, since it dramatically slows the growth of organisms which might contribute unpleasant flavors. Ideally, the ferment should not be allowed to go below pH 3.4 nor above 4.0. Judicious use of potassium or calcium carbonate or bicarbonate will allow you to keep the pH in this range (potassium carbonate is more soluble and easier to work with, calcium carbonate is more widely available). Both sodium carbonate (washing soda) and bicarbonate (baking soda) will work, but are not as beneficial to the yeast as the potassium or calcium compounds

3. Not enough oxygen for the growth phase of the yeast

   Yeast needs oxygen early in the fermentation process to grow and to produce lipids, which protect the yeast cells from high alcohol levels later in the fermentation. Frequent agitation or aeration of the fermenting fluid during the first 24-36 hours will supply enough, but oxygen should be excluded after this time by use of an airlock.

4. Too much carbon dioxide in solution in the fermenting liquid

   Carbon dioxide is a waste product of fermentation, and at high concentration can slow down the fermentation, especially when the yeast is already stressed by other factors. Occasionally agitating the fermenting fluid will release the excess, as will the addition of powdered materials (nutrient or potassium carbonate additions). This can result in the production of considerable foam, so be careful!

Yeast re-hydration

Active dry yeast is often not properly re-hydrated. The first few seconds of re-hydration, as the cell walls re-constitute themselves, are critical to the survival of the yeast. Re-hydrating at too low or too high a temperature will cause a large percentage of the added yeast to be sickly or die, resulting in slower than expected fermentation. The same effect is caused by excessively high sugar concentrations at the beginning of the fermentation. This can cause up to 80 % of the added yeast to be unhealthy and die early in the fermentation.

Proper re-hydration technique for active dry yeast is:

- 1 part by weight to 5 parts sterile (boiled) water (e.g. 50 gm yeast is re-hydrated in 250 ml. Water).
- Initial water temperature 38º - 41º C (100º - 105º F)
- Mix well, and allow to sit for at least 10 and not more than 20 minutes before adding to ferment.

CAUTION! This procedure is for pure, active dry yeast, and should not be followed for yeast products that contain nutrients. These are formulated for specific method of use, so be careful to follow the package directions.

Sugar management

Controlled fermentation requires control of the initial sugar concentration. Assuming that fermentation is about 90 % efficient, it takes about 17grams of sucrose per liter of solution to produce 1.0 % alcohol by volume. Some fermentations will do better than this, and some worse, but it is a decent “rule of thumb” to use with pure table sugar. Glucose or Fructose require 17.9 grams per liter per percent alcohol. This is explained in Appendix 2.

Most home brewers or wine makers have a hydrometer to measure sugar content in their wort or must. Many of these hydrometers have several different scales on them. There are many possible scales for measuring the density (and hence, the sugar content) of a solution. The most common include: Specific Gravity and degrees Plato, Brix or Ballings.

The specific gravity of a solution is the ratio of its density to that of pure water. A specific gravity of 1.050, for example is 5 % denser than pure water. The Plato, Brix and Ballings scales all relate the density of the solution to the % sucrose by weight. Sucrose (common table sugar) was chosen because it is the sugar that dissolves in water to produce the greatest density. Appendix 3 includes a table that shows the relationship between sugar content and several common hydrometer scales.

Fortunately, we do not need a hydrometer to make up a correct sugar solution for fermentation. All we need to do is calculate the grams per liter of sugar we want from the % alcohol we're trying to reach using the following equation:

- % alcohol desired x 17 = grams of sugar per liter of solution

Then, calculate the weight of sugar needed based on the g/l sucrose desired, using one of these equations:

- **Metric units:** g/l sucrose desired x liters desired ÷ 1000 = kg sugar
- **American units:** g/l sucrose desired x US gallons desired ÷ 120 = lb. sugar
- **Imperial units:** g/l sucrose desired x Imperial gallons desired ÷ 100 = lb. sugar

BE CAREFUL! You are after g/l of sugar in the **final solution**, so weigh out the sugar and slowly add enough water to end up at your final amount of liquid! If you add the sugar to the measured amount of water, you will end up with more liquid that is less concentrated than you expected. *If you use some sugar other than sucrose, you can dissolve the proper grams into the correct final volume, but the specific gravity or degrees Plato measured with a hydrometer will mislead you.*

Here are some sample calculations to show the various ways these equations can be used:

- **We want to end up with 9 US gallons of 20 % alcohol. How much sugar do we need?**

  20 % alcohol x 17 = 340 g/l sugar.

  340 g/l sugar x 9 gallons ÷ 120 = 25.5lb sugar

- **We have a 25 pound sack of sugar. How much 15 % ethanol can we make?**

  15 % alcohol x 17 = 255 g/l sucrose

  (now, turn the equation around. Volume x g/l ÷ factor = Weight Sugar, which means that Volume = Weight sugar ÷ g/l sugar x factor. We'll work this all three ways.)

  Metric: 11.34 kg ÷ 255 g/l sugar x 1000 = 44.5 liters

  American: 25 lb ÷ 255 g/l sugar x 120 = 11.76 US gallons

  Imperial: 25 lb ÷255 g/l sugar x 100 = 9.8 Imp. Gallons

  (As a practical matter, rounding to the nearest quart or liter is just fine)

- **We have 10 kg of sugar, and a 35-liter fermenter. What % alcohol will we get?**
  (This time we know weight of sugar and volume. Re-arrange the equation again to get
  g/l sugar = weight sugar ÷ volume x factor
  = 10 kg ÷ 35 liters x 1000 = 28.6 % sugar
  (now, calculate % alcohol from g/l sugar, by dividing by 17)
  % ethanol = g/l sugar ÷ 1.7
  286 g/l sugar ÷ 17 = 16.8 % ethanol

Dr. Cone's methods for high alcohol fermentation

Method #1

This method uses a lot of yeast, because about 80 % of it will die during the first day or so of fermentation due to the high sugar content.

- Start with sugar concentration of 400 grams per liter of solution.
- Add 1/2 gram Fermaid K® (or equivalent) per liter (2 grams per US gallon).
- Add at least 2 grams properly re-hydrated yeast per liter (8 grams per US gallon).
- Add 1/8 gram potassium or calcium carbonate per liter (1/2 gram per US gallon) initially.
- Add 1 gram DAP per liter (4 grams per US gallon) divided into at least three doses, spread out over the first half of the fermentation.
- Ferment between 21º and 26.5º C (70 – 80º F).
- Aerate, stir or pump around for the first 48 hours.
- Monitor the pH carefully, keeping it between 3.4 and 4.0. Add potassium or calcium carbonate as needed to keep in this range.
- Stir occasionally after fermentation slows down to keep the yeast suspended.

Method #2

This method starts with lower sugar content and less yeast, and relies on growing your own yeast during the fermentation. Since yeast can be expensive, this is the less expensive method, but it requires more work and monitoring than method #1.

- Start with sugar concentration of 160 – 220 grams per liter of solution.
  When sugar content drops to about 41 g/l (1.016 s.g., 4º Plato), add 20 grams per liter (2-1/2 oz. per US gallon). Continue this cycle until you have used all the sugar planned for the alcohol content desired.
- Add 1/2 gram Fermaid K® (or equivalent) per liter (2 grams per US gallon).
- Add 1/2 gram properly re-hydrated yeast per liter (2 grams per US gallon).
- Add 1/8 gram potassium or calcium carbonate per liter (0.5 g per US gallon) initially.
- Add 1 gram DAP per liter (4 grams per US gallon) divided into at least three doses, spread out over the first half of the fermentation.
- Ferment between 21º and 26.5º C (70 – 80º F).
- Aerate, stir or pump around for the first 48 hours.
- Monitor the pH carefully, keeping it between 3.4 and 4.0. Add potassium or calcium carbonate as needed to keep in this range.
- Stir occasionally after fermentation slows down to keep the yeast suspended.

Turbo yeast products

Several different suppliers provide “Turbo” yeast products (many of which were designed by Dr. Cone), which contain carefully selected strains of yeast combined with nutrient and buffering mixtures designed for fermenting plain sugar. They are claimed to be able to ferment to 12-14 % alcohol in as little as three days, and some of the newest ones claim to be able to ferment to 18-20 % alcohol in as little as a week. Some new "Turbo" products have been designed to ferment at quite high temperatures, which is good news for those living in hot climates!

We have tried a few of them, and the results are quite good, though not quite as fast as claimed. While maintaining temperatures at the low end of the recommended range to minimize congener production, we have achieved 20 % alcohol in 12 days with an “8 kg” "Turbo" and 12 % alcohol in 5 days with a “6 kg” turbo (the "8 kg" and "6 kg" refer to the manufacturer's recommended quantities of sugar to use with each packet of "Turbo" yeast). In both of these cases, another two to three days were required for all the yeast to settle to the bottom of the fermenter.

The advantage of the "Turbo" products is that they require very little supervision while they are working. All the yeast, nutrients and buffering compounds are in a single packet. The one problem is that they can be so effective that the fermenting wash can heat to the point of killing the yeast, especially if an oversized batch is attempted. Techniques for lowering the temperature may have to be employed to ensure success.

Techniques for employing either the Cone Protocol or turbo yeast

The turbo yeast packets are generally sized for a fermentation volume of 25 liters (6.6 US gallons), too large to fit in a standard carboy. The Cone methods may be employed on any volume from one gallon to 10,000 gallons or more, provided the equipment is capable of handling the tasks of oxygen, pH and temperature management.

For the home producer, 25 – 50 liters (6 – 13 US gallons) is a very reasonable volume to work with.

The easiest technique is to utilize one of the large plastic fermenters normally used for primary fermentation of wine (a plastic trash can with a lid). As noted earlier, the biggest single drawback to these fermenters is that if they become scratched (from over-vigorous agitation or stirring, for example), they can become very difficult or impossible to sanitize adequately. Fortunately, they are cheap.

A simple way to manage the temperature of a fermentation in one of these containers is to place the fermenter in a laundry tub connected to hot and cold water and a drain. Add the sugar to the fermenter, dissolve it in a small amount of hot water, and bring it to the desired volume and temperature with cool water. Then fill the tub around the fermenter with water at the desired temperature. The extra thermal mass of the water around the fermenter will greatly slow down any temperature swings or spikes created by active fermentation. A liquid crystal thermometer placed on the fermenter (above the level of the water around the fermenter, but below the level of the fluid inside the fermenter) will allow you to monitor the temperature. If it starts to climb, drain out some of the water in the tub and replace it with cold water. One or two adjustments should be all that is required during a fermentation.

Another method of controlling temperature is to freeze water inside of sanitized plastic bottles or jugs, and then place one or more of these jugs directly in the fermenting wash. Experience will teach what size of bottle works best and how many it takes to drop the temperature a specific amount. You could also construct a cooling coil out of copper or stainless steel tubing and run cold water through it. Copper will be etched by the acidity of the fermenting fluid, so a copper coil should NOT be left in the fermenter for a long time. Just put it in when the temperature needs to be reduced, and remove it and rinse thoroughly as soon as the temperature is reduced to the desired level.

Fig. 1-4

For more active control, a system with aeration, circulation and temperature control can be devised. One old idea in this direction is to use a laundry tub as a fermenter, and place an aquarium pump and heater in it. This idea will work, but a plastic laundry tub has all the same problems of sanitation as the trash can, and costs considerably more to replace.

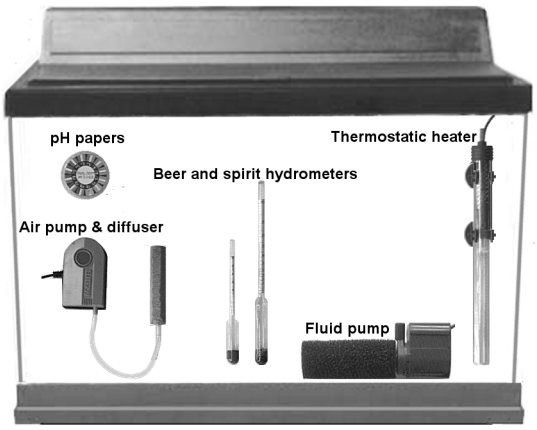


Fig. 1-5

We suggest that if you want to experiment with active systems and controls, you purchase a 75-110 liter (20-30 US gallon) aquarium starter kit. These can be obtained inexpensively at almost any tropical fish or pet store. A starter kit usually comes with the aquarium, a circulating pump, an air pump, a heater, a thermometer, and a sliding or folding glass lid for the aquarium. The only thing you will have to add is some means of temperature reduction – such as frozen plastic bottles or a cooling coil.

You can buy everything you need in one place at a low price. Glass is easy to clean, and it's easy to watch the process through the sides of the aquarium. For example, you can see if yeast is settling, which might indicate that you should change the circulation pattern to avoid dead spots, or stir the tank a bit. After the fermentation is complete, switch off the pump and heater, wait for the yeast to settle out, and siphon the fermented fluid away from the settled yeast.

CHAPTER 2

HOW DISTILLATION WORKS

Vapor pressure

Every substance is a collection of atoms and/or molecules held together by mutual attraction. The temperature of that substance is a measure of the kinetic energy these molecules have - the faster they move and vibrate, the higher the temperature. Depending on the temperature and the pressure, the molecules may pack together tightly as a solid, may pack loosely as a liquid, or may freely move around as a vapor. These different states of matter are called phases, and the transition from one phase to another involves the absorption or release of large amounts of energy in the form of heat.

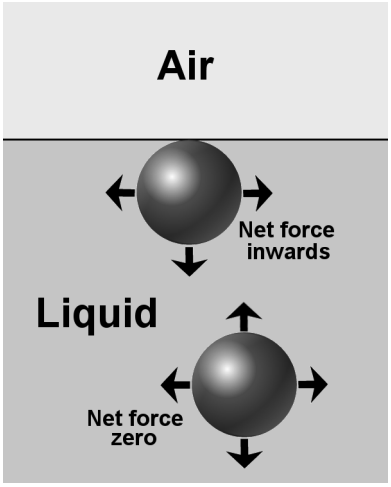


Fig. 2-1

Molecules can escape from a solid or liquid substance to form a gas. When they escape from a solid, this is called **sublimation**; escape from a liquid is called **evaporation**.

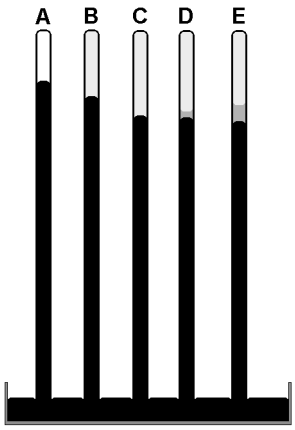
Molecules manage to escape by having enough energy to get through a barrier at the surface of the substance. This barrier is created by the fact that the attractive force between molecules is directed inwards at the surface of a substance. Inside the body of the substance, the attractive forces are arrayed in all directions, and cancel themselves out.

This is a very strong force in liquids and is called **surface tension**, meaning that liquids act as if they have a "skin" on their surface holding them in. Surface tension causes liquids to form a meniscus against the side of a glass (the curved bit 'sticking' to the sides), and makes globules of mercury scoot around like ball bearings. Surface tension is strong enough to allow many insects to walk on water!

It takes a lot of energy to vaporize a liquid, far more than to just heat it up, and the extra energy is needed to overcome the attraction between the molecules in the liquid. The stronger the attractive force between the molecules of a liquid, the harder it is to vaporize. Once molecules leave the liquid or solid, their rapid motion adds to the pressure of the environment. Each substance produces a well-defined amount of pressure at a given temperature, known as its **vapor pressure**. The total pressure of the surrounding environment is made up of the pressure contributed by all the substances present.

Do not confuse vapor pressure with smell! It is true that scents and smells come to us by way of airborne molecules, but a strong smell does **not** imply a high vapor pressure. The strongest smelling substances of all are a group of chemicals called mercaptans, which include the famous "eau de skunk". As powerful as the stink is, though, it takes very, very few of these molecules to empty a room. The tiny number of molecules required to overpower your nose exert a negligible vapor pressure. More to the point of distilling, the almost odorless ethanol has a higher vapor pressure than the heavier fusel alcohols that appear late in a run, and which smell awful.

An illustration of vapor pressure



The vapor pressure of a substance is the amount of pressure escaped molecules can exert on the surrounding environment. A simple experiment often performed in schools illustrates this. Warning! Mercury is toxic; please do not attempt this experiment yourself at home.

Take a long glass tube that is closed at one end, and fill it with mercury. Upend it with the open end submerged in a beaker of mercury. If the tube is long enough, an empty space will appear above the mercury in the tube, and the mercury column will only be about 760 mm (30 inches) high in the tube (tube A). This empty space is a vacuum, and it's there because the weight of a mercury column this high just balances the pressure of the atmosphere. If we filled the tube with water, the liquid would be about 9 meters (30 feet) high!

Fig. 2-2

Now introduce a few drops of water into the bottom of the tube. They will float to the top of the mercury and boil rapidly because they are now in a vacuum. At first, each droplet will boil off completely and the level of the mercury will fall, because the water vapor created by the boiling droplets exerts pressure on top of the mercury (tubes B and C).

Eventually, you will be left with a few drops of water floating quietly on top of the mercury. By this time the mercury column will have lowered by about an inch or two. Adding more water will only result in a layer of water on top of the mercury, but the open area above the liquid will not increase in size (tubes D and E).

The lowering of the mercury column directly reflects the vapor pressure exerted by the water vapor. Every substance can only exert a certain amount of vapor pressure at a given temperature. In a confined space, there is a maximum concentration of molecules of a substance that may be present as vapor. When this point is reached, the vapor is called **saturated**. In a saturated vapor, molecules are condensing from vapor to liquid as rapidly as they are vaporizing. The pressure at which this occurs is called the Saturated Vapor Pressure, and there is a characteristic saturated vapor pressure for every compound.

If you increase the temperature of the apparatus, the water droplets will begin to boil again and the mercury level will lower even more. At 100 $^{\circ}$C (212 $^{\circ}$F) the tube will contain only saturated water vapor, because the vapor pressure of water at this temperature equals the pressure of the surrounding atmosphere. This is the definition of the **boiling point** of a substance.

If you lower the surrounding pressure, you lower the boiling point. Increase the surrounding pressure, as in a pressure cooker, and you raise the boiling point. This is very important to the process of distillation. If you are interested in exploring this further, there is a comprehensive explanation in Chapter 8, where we be show in detail how you can use this knowledge to calculate precisely the concentration of ethanol you will get at each stage of distillation.

Boiling

Water droplets above the mercury column boiled at temperatures well below 100ºC, because the pressure was much lower than normal. Evaporation from the surface of a liquid is a slow, stable process that occurs at all temperatures. Boiling is a phenomenon that occurs when the vapor pressure of the liquid exceeds the pressure inside the liquid and bubbles form. In boiling, evaporation happens **within** the liquid and not just from its surface. The bubbles create new surfaces throughout the body of the liquid.

Since the pressure within the liquid is slightly higher than that at the surface, because of the weight of the liquid itself, the bubbles expand as they rise, which makes boiling more dramatic than evaporation,

though it is really same process. In both evaporation and boiling the same amount of energy is required to overcome surface tension, wherever that surface may be – on top of the liquid or surrounding a bubble. Boiling speeds up evaporation by producing a more surfaces, but the same amount of energy is needed for each molecule to escape from the liquid and enter the vapor phase.

The scientific measure of heat energy is the **calorie**, which is the amount of heat required to raise the temperature of one gram of pure water by 1ºC. A thousand calories is called a Calorie (with a capital C) and this is what we use to measure the energy contained in food or expended in exercise. It takes 80 calories to heat one gram of water from 20ºC (68ºF) to the boiling point of 100ºC (212ºF), but it takes 540 calories to turn that same gram of water into vapor without raising the temperature! The surface tension of water may not seem like much to us, but for molecules, its a **huge** hurdle to jump!

Physical constants like the boiling point of a liquid are measured or defined under standard conditions. When a liquid is heated, it is possible to **superheat** it, or raise its temperature above the boiling point, without boiling occurring. This is because it takes more energy to **create** a bubble than to simply evaporate into one. Once the liquid is boiling, the bubbles that are already formed trigger the formation of others.

Superheated liquids can **bump** or suddenly erupt into frothy boiling, also known as **surge boiling**. Placing materials with sharp corners and edges into the liquid before heating it can prevent this by promoting the formation of bubbles, which allows boiling to begin in a gentle and controlled fashion. **Boiling chips** are used in laboratories for this purpose, but we have found that ordinary plain metal pot scrubbers do an excellent job as well. Surge boiling is discussed in more detail in Chapter 4, as a factor in boiler control.

Mixtures and solutions

All substances are made up of atoms and molecules that contain electrical charges. Depending on the type of charge and the way it is distributed, substances can attract or repel one another very much, very little, or not at all.

When substances are mixed together, different things can happen depending upon the nature of their interaction. If the molecules actively repel one another, like oil and water, they will separate from one another as completely as possible and are said to be immiscible. These substances may be easily separated by mechanical means - filtering in the case of a solid and a liquid (sand and water, for example), or siphoning off in the case of two liquids (the previously mentioned oil and water). There is an exception to this rule: when the immiscible components are made into extremely small particles, the motion caused by thermal energy keeps re-mixing them so they can't separate themselves. This special case is a process called homogenization, but it does not affect the process of distillation.

If the molecules are strongly attracted to one another, they can chemically react to form a wholly new and different compound, consuming the original substances in the process. The metabolism of living things is a series of controlled chemical reactions, and a good example of this process is the conversion of sugar to carbon dioxide and ethanol. There is no way to get the original sugar back, because it no longer exists.

If the attraction between molecules is moderate, the substances can go into solution. In a solution, the molecules of the two substances are intimately mixed together, and there is no way to separate them by mechanical means like filtering or centrifuging. However completely mixed they are, they still exist and maintain their unique properties, like vapor pressure.

Water is a good solvent for many different things, and the complex composition of seawater or blood is a testament to this. Salts and most (but not all!) solids have a vapor pressure so low that we can consider it to be zero. If you dissolve a teaspoon of salt in a cup of water, and then allow the water to evaporate, you will end up with a cup containing a teaspoon of salt. The water evaporates, but the salt doesn't. Boiling the water speeds things up, but the end result is the same.

The same is true of oil dissolved in gasoline. Oil and gasoline are chemically similar molecules, but the oil molecules are larger and have a much lower vapor pressure than gasoline. If you want to put a

very thin film of oil evenly over a component, dissolve the oil in gasoline and dip the component in it. When the gasoline evaporates, you're left with a very thin, even coating of oil all over the component (this is an old watchmaker's trick!)

It is relatively easy to separate the components of a solution when their vapor pressures are quite different. What happens when you try to separate components with similar vapor pressures?

Pure water is a liquid that boils at 100ºC (212ºF) at normal atmospheric pressure (760 mm mercury) and has a surface tension of 54.9 dynes/cm^2 (a **dyne** is a unit for force, defined in Appendix 1). Pure ethanol is a liquid that boils at 78.5ºC (173.3ºF) and has a surface tension of 21.38 dynes/cm^2. You've probably guessed that the ethanol has a **higher** vapor pressure than the water, because it has a lower surface tension. This is true, but they are much closer than salt and water or oil and gasoline.

Now mix water and ethanol together, like you did with the water and salt, and try to separate them by evaporation. What do you get? An empty cup! If you leave the mixture for a limited time, the more volatile ethanol does evaporate faster than water. You would get a cup containing a little water, but if you collected the vapor coming from it, you would not have pure ethanol. You would have a mixture of water and ethanol. Ethanol and water are much more difficult to separate than water and salt or oil and gasoline.

You can try to hurry things along by heating the mixture to a temperature above the boiling point of pure ethanol but below that of water, on the assumption that the ethanol will boil off but the water won't. Right?

Wrong! Many people have tried this, they've all been beaten by the laws of physics. As you heat the mixture up, ethanol molecules will escape faster than water molecules, but **both** will still escape. You may end up with a little pure water in the bottom of the cup, but the vapor will be a mixture of ethanol and water. However, this idea is getting closer to the solution.

What happens if you boil the mixture as rapidly as possible? Exactly the same as before, but faster! After all, boiling is really just fast evaporation.

To make sense of this, we need to examine the behavior of mixtures. In general, the boiling point of a solution is found somewhere between the boiling points of its components. What allows the distillation process to work is the fact that a mixture boils at a temperature that depends upon the relative concentrations of the components of the mixture, and produces a vapor that is a mixture of the two substances. The vapor produced is not just any mixture, but a predictable one. At any given temperature, the substance with the higher vapor pressure produce more vapor than the less volatile substance.

For ethanol and water, this means that you end up with a vapor containing a higher proportion of ethanol molecules than the starting mixture. Excellent! You can now condense this vapor and enjoy a good drink, which is exactly what they did in the Good Old Days. It shouldn't take long to figure out that repeating the process again provides something even richer in ethanol, and so on…

What we've just described with water and ethanol applies to many other substances, including the congeners mentioned in chapter 1. Some of them have higher vapor pressure than ethanol, and some lower. Some of them provide flavor to the spirits, and some create headaches. Repeated distillation increases the concentration of ethanol, but also alters the concentration of other substances in the mixture. The end result is more ethanol with less flavor and fewer hangovers.

Two basic approaches evolved to deal with this limitation. Spirits produced in bulk were distilled two, three, or even four times, and then either **treated** or **matured** (more on this later) before being consumed. This was fine for an ongoing commercial operation, because production from previous years was available while the fresh spirits aged.

On a smaller scale, and often for the production of medicines and tonics, the ethanol was repeatedly distilled to make it as pure as possible. At the same time, botanicals were steeped or distilled for their flavors and added to the purified spirits. These extracts were then blended with other substances to produce the exact flavor, color and palate desired. In general, these products required less maturation than their bulk cousins, but they often benefited from it, because some subtle flavors can't be produced in any other way.

These herbal tonics and medicines were often produced as a holy calling by monastic orders. The brothers had the combination of herbal, medicinal and process knowledge, the space to cultivate and protect the rare herbs, and the time to perform the difficult processes of extraction and purification.

To complete the discussion of concentrating ethanol by repeated distillation, we must consider **azeotropism**. By repeatedly boiling and condensing a mixture of water and ethanol, we get distillates that contain higher and higher proportions of ethanol. This works until the solution contains about 96% ethanol and 4% water. At this point, the proportions of ethanol and water molecules entering the vapor phase remain the same as the proportions in the solution, 96% and 4%. This is caused by the extra attraction of the molecules in solution, and defines the upper limit of concentration by distillation. Another result of azeotropism is that the boiling temperature of the 96% ethanol solution is lower than that of pure ethanol!

To obtain 100% pure ethanol (something only chemists and alternative fuel enthusiasts need), you have to "break the azeotrope", which requires adding a third substance to disrupt the molecular attraction between water and alcohol. Due to the principles of distillation, this substance will also appear in the mixture of vapors. The most effective breaker of the ethanol/water azeotrope is benzene, which is **highly toxic**

Fortunately, you don't need to bother. 96% is an excellent solvent for making extracts and essences, and if you're going to produce a 40% vodka, why take all the effort to remove the water only to put it back?

A graphical description for mixtures

A picture is truly worth a thousand words, and a few graphs can help create a clear understanding of what happens when mixtures are heated and condensed. These phenomena are the reason that distillation works, and understanding them is essential to designing any type of effective distillation apparatus.

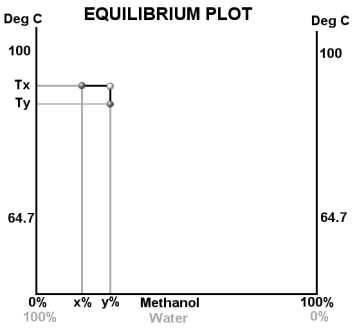


Fig. 2-3

Let's start with a methanol/water mixture with X% methanol by volume. We've chosen methanol because its characteristics are similar to ethanol, but they're not complicated by azeotropism. (Methanol is **not** what you find in bottles marked 'methylated spirits'. That is ethanol, which has been made poisonous by adding small amounts of methanol and other noxious substances that cannot be removed by distillation).

This chart plots the boiling points of solutions of methanol and water as a function of the methanol content.
The top left dot shows that the boiling point of a solution containing X% methanol is TxºC. The vapor produced contains a higher percentage of methanol, because it has a higher vapor pressure than water (shown by the top right dot at TxºC) and this vapor will condense at TyºC.

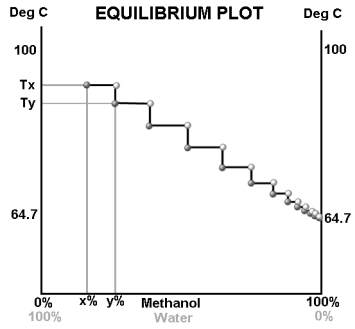


Fig. 2-4

Subsequent vaporizations and condensations are plotted in this chart. As the concentration of the condensed liquid approaches 100% methanol, the boiling point 64.7ºC.

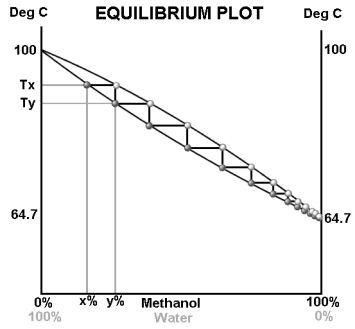


Fig. 2-5

Joining up the dots produces two separate curves. The upper one is called the **vapor line**. Any point above it is vapor, and anything below the lower line, the **dew line**, is liquid.

Points lying in between the lines represent a dynamic mixture of liquid and vapor. As a droplet of liquid starts at the dew line and slides towards the vapor line to its right at a constant temperature, it gradually grows smaller as it transforms into a vapor. The liquid and vapor in such a situation are said to be **in equilibrium**, and charts like this one are called **Equilibrium Charts**.

Plates

An area where vapor condenses, hangs around and then vaporizes again is called a **plate**. This name comes from the fact that commercial fractionating towers for oils and fuels contain trays or plates to hold liquids while the surrounding vapor is brought into intimate contact with them. Each of the horizontal lines in Fig. 2-5 can be considered a "plate". Of course, these "plates" are imaginary, and will move around depending on where you started on the curve.

The effect of Azeotropism

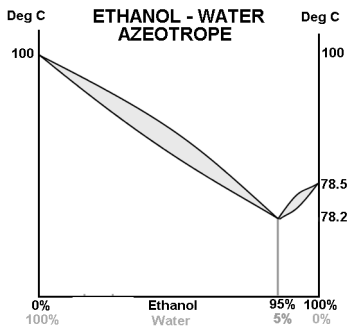


Fig. 2-6

The equilibrium chart for methanol/water solutions is quite simple. The case of ethanol/water solutions is more complicated, with a kink at the bottom of the curves. This kink is caused by the azeotrope formed by ethanol and water at about 96% ethanol. Vaporization of liquid at this concentration yields vapor with a the same composition as the liquid. If you vaporized a mixture stronger than 96% ethanol, the concentration of ethanol in the vapor would be **less** than that in the liquid and, once again, the system would settle at its azeotropic point.

Distillation alone at atmospheric pressures cannot give a concentration of ethanol higher than 96%. Distillation under a vacuum will, but this requires special apparatus. Fortunately, 95% ethanol is more than sufficient for our needs.

Chemists have derived equations to calculate of the number of theoretical plates needed to achieve any desired degree of purity in a distillation. When these calculations are performed for ethanol and water, it turns out that 95% ethanol requires at least 12 plates under full **reflux** conditions (see the next chapter for a discussion of reflux). We won't trouble you with the calculations here, but we'll discuss them in detail in Chapter 8.

We've described what happens with a mixture of two substances in order to keep the graphs simple, but the same principles apply to mixtures of three or more substances. The key point is that the higher the vapor pressure of a substance, the easier it is for its molecules to move from the liquid to the vapor phase, and the greater its proportion in the vapor.

Cleaning and Polishing

We mentioned earlier that the 'tasty stuff' in flavored spirits such as rum or whiskey is derived from congeners present in the fermented mixture. Unfortunately, the congeners are not very pleasant when fresh - in fact, they can be downright horrible or even poisonous! These congeners include **fusel alcohols** (from the German "Fusel", or "bad alcohol"), oils and volatile esters. Some of these substances contribute nicely to flavor and bouquet, but most do not. Some of them are the major source of hangovers!

Carbon treatment of raw spirit can remove many congeners, both the pleasant and the unpleasant ones. Many brands of commercial spirits are filtered through charcoal, or stored in charred wooden barrels, often with the claim that this produces a better-tasting product.

Chemists have discovered quite a bit about the manufacture and use of **activated carbon** for adsorbing chemical contaminants, and we can use this to profitably. (Note the word **adsorption,** not absorption. **Ab**sorption is what sponges do when water is carried bodily in the holes inside the sponge. **Ad**sorption is where individual molecules of a contaminant are held onto the inside surface of the porous carbon by electrostatic attraction or by loose chemical bonding).

Activated carbon products are carefully designed for their end use, and manufactured in a process which involves careful selection of ingredients, very high temperatures and gas or steam treatment. They work by physical adsorption of contaminants onto the enormous internal surface area of the carbon, typically 1,000 square meters (a quarter of an acre) per gram (hard to believe, but true!). Remember, it's a physical and not a chemical effect that makes them work.

It pays to be very careful in choosing the source and type of activated carbon you use to clean spirits. Aquarium carbon will **not** do! This is a cheap mixture of many sorts of charcoal that may remove some of the congeners from the spirit, but can also introduce nasty trace elements and flavors of its own. Fish don't mind, but you will! Properly made activated carbon is on the market now, specifically designed for the purpose of cleaning spirits - a process called **polishing**. More information about activated carbon is in Appendix 4.

Removing the contaminants is a rapid and effective method of dealing with congeners, but one that produces flavorless spirits. These spirits are usually then mixed with flavorings to produce a wide variety of beverages

Maturation

Just as wines generally benefit from storage and aging in wooden barrels, so do spirits, especially when we desire the flavor of the original fermented product, as in rum, whiskey and brandy. Wooden barrels allow small and controlled amounts of oxygen to reach the contents. This oxygen, along with substances extracted from the wood, allow many complex chemical reactions to occur. Acids, alcohols, tannins and other substances react together over time to produce a wide variety of new compounds. The long-chain alcohols present in the fusel alcohols are transformed to complex esters and other compounds that add flavor and are not as hard on the body.

These processes take time. The actual amount of time is partly a function of the size of the barrels used – larger barrels take longer, because there is less surface area per volume contained. This means it takes longer to extract enough compounds from the wood and for enough oxygen to get through the walls of the barrel.

This maturing process has nothing to do with some strange ability of congeners to 'seep' through the wood and disappear from their confinement. Some people in the past have seriously claimed that fusels can do this because they are 'slippery oils'! This misunderstanding highlights the need for caution when reading old manuals for the production of beverages – often the authors did not understand the nature of the processes they used.

CHAPTER 3

PUTTING THEORY INTO PRACTICE

In Chapter 2, we showed you how distillation works, and now we'll start to apply this knowledge. First, let's define some terms to make sure we are all speaking the same language.

Batch and Continuous Distillation

These terms don't require any special definition, because they mean exactly what their names imply. Continuous distillation is characteristic of large, commercial operations, whether oil refineries or gin distilleries. The process just go on and on as crude oil or fermented brew is fed into the input and a distilled product is continuously withdrawn from the output.

Batch distillation is characteristic of smaller scale production. Small quantities are dealt with one at a time, and this is the process that we will be discussing. Commercial whiskey production is an interesting mixture of the two methods: large quantities are processed, but in a manner reminiscent of small batch stills. There's a reason for this, and we'll come back to it later.

Reflux

Reflux is a term that is often used in a confused or incorrect manner. **Reflux** is nothing more than vapor that condenses inside the still and drips back toward the boiler. Refluxing is an important component of most distilling processes.

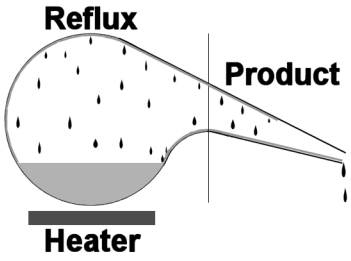


Fig. 3-1

Fig. 3-1 shows reflux occurring in a laboratory retort, about the simplest still imaginable.

Reflux and Product Ratios

The **Reflux Ratio** is the ratio of the vapor returned to the still as reflux to **the total amount of** vapor produced. The reflux ratio is often expressed as a percentage: if the boiler produces 100 parts of vapor and 90 parts of this are condensed and returned, then the reflux ratio is 90%. Operating under 100% reflux, or at a reflux ratio of 100%, means that **all** the vapor is condensed and returned to the still.

The ratio of the amount withdrawn as product to the total amount of vapor is the **product ratio**, or "draw down" of the system. It is sometimes confused with reflux ratio. A simple guide to keeping these terms straight is "Product goes out, Reflux goes back".

Distilling water

The simplest distillation you can perform is to heat up water and then condense the vapor. This results in a distillate that contains only water, and nothing else. It will contain no salts or any other non-volatile dissolved substances. Many people distil water at home, using a variety of small commercially produced water distillers. Industrially, enormous plants produce millions of gallons of distillate from seawater for irrigation and drinking water

Textbooks often present the classical **survival still** as a way to survive in the desert by distilling contaminated water. Let's discuss this simple but very inefficient design, and see how it can be improved.

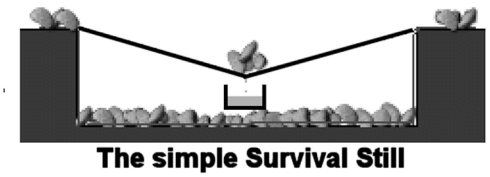


Fig. 3-2

Fig. 3-2 is a diagram of the simple survival still. It is merely two sheets of transparent plastic, one held down at its edges by stones and stretched over a pit lined with the other. Stones are put in the middle of the sheet to create a low point, and a pot or other collecting vessel is placed under it. Contaminated water or moist material, like plants, is placed in the pit around the collecting vessel.

Sunshine passes through the transparent sheet and warms up the stones and liquid in the bottom of the pit, causing the contaminated water to evaporate. This vapor condenses on the sheet and drips from the low point into the collecting vessel. A good yield might be a couple of mouthfuls of distilled water each day, which is not enough to keep you alive in a hot desert.

This is a very small yield, especially with the considerable energy pouring down from the sun in desert areas. What's wrong with the design?

Actually, the flaw in the design is quite simple. The large quantities of water evaporate, but the condenser is a sheet of plastic sitting in the hot sun! The only cooling available comes from the wind, which may or may not blow. No wonder very little finds its way into the collection vessel, and most of the hot vapor escapes.

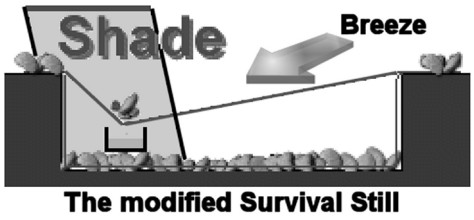


Fig. 3-3

One simple improvement, shown in Fig. Fig.3-3, is to separate the heating action from the cooling. One way of doing this is to place the stones at one side, rather than in the middle. Then shade the side with the stones. The condenser will still be warm, but a lot cooler than if it were in the direct sunlight, which is now confined to the heater side. The larger or darker the shaded part, the cooler it will be.

If you have some contaminated water to spare (use your imagination on where that might come from!), you can make it even more efficient by moistening a thin layer of soil sprinkled on top of the "condenser" area of the sheet. Evaporation of this water will help cool the condenser sheet and produce even more drinking water inside.

That's just one way of improving the survival still. There are many other ingenious ones, and you should spend some time thinking about how you could make it more efficient (try a metal plate lying between the plastic and the weighting stones above the cup). This time will be well spent, because it will firmly fix the principles of distillation in your mind. Even very simple systems may be improved if you understand the process that's taking place, and you're not afraid to experiment.

Boilers

Fig. 3-4

Every practical still needs a boiler, and all boilers have the same basic characteristics: a well-sealed container with a way of heating the contents and an outlet for the vapor. Successful boilers can be made from household pots and pans, domestic water heaters, and a wide variety of other devices.

The earliest stills were ordinary pots or cauldrons fitted with a snugly fitting lid called an **Alembic**. The alembic featured a spout that directed the vapor to one side so that it could be condensed and the product collected. This proved to be such an efficient design that it's still used today in many laboratories. A diagram of a modern laboratory retort was used earlier to illustrate reflux. The similarities between the old alembic shown here and the modern retort are obvious.

The size of the boiler doesn't matter much, except for the time needed to bring its contents to a boil, but control of the heat put into it does. We will discuss this in detail later. A well-insulated boiler is more efficient and easier to control.

Boilers generally fall into one of two groups: **directly** and **indirectly** heated.

Direct heating

Direct heating features a hotplate or heating element in close proximity to or even inside the boiler. Three common modes of direct heating are the hotplate, the concealed element and the immersion element.

The Hotplate

Fig. 3-5

A hotplate is an external device used for heating a pot or kettle. (We would include the burner on a kitchen stove in our definition of a hotplate). Hotplates may be fired by gas or electrically heated. There are a wide variety of hotplates available, and in general you get what you pay for.

The "Concealed" Element

Fig. 3-6

The concealed element is mounted within the bottom of the boiler, and is therefore "concealed" from view. The bottom of the boiler is often thick, functioning as a built-in heat diffuser. These elements are common in consumer electric devices, like coffeepots, teakettles and deep fryers, where they work very well. Since the element is not immersed in the liquid, it's relatively easy to incorporate circuitry to protect the element should the boiler run dry.

The Immersion Element

Fig. 3-7

An immersion element is mounted through the side of a boiler and is in direct contact with the liquid. Electric water heaters use immersion elements. If the element is larger than needed, it will require direct and sophisticated power control to avoid surge boiling. A thermostat in the boiler will **NOT** prove satisfactory. (Thermostats work in water heaters because they operate well below the boiling point).

Indirect Heating

An indirectly heated boiler is supplied with heat that is generated elsewhere, and then transferred to the boiler in a well-controlled manner. Frequently, the laws of physics control the temperature and the rate of heat transfer (for example, you cannot provide more than 100ºC with hot water. This prevents burning in cooking, and is particularly suitable for processing botanicals or thick mashes, which might be ruined by higher temperatures). The two main methods of indirect heating are jackets and coils.

The Bath or Jacket

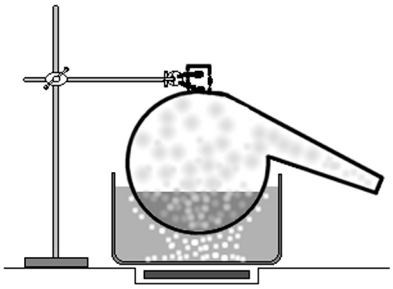


Fig. 3-8

The boiler is held inside another container that holds heated liquid or steam. The arrangement can be two separate pots (like a double boiler), or a shroud or channel permanently mounted to the outside of the boiler. The jacket can be directly heated itself (again, like the common double boiler), or fed with hot liquid or steam from a separate boiler. Jacketed heating vessels are very common in industry.

Indirect heating

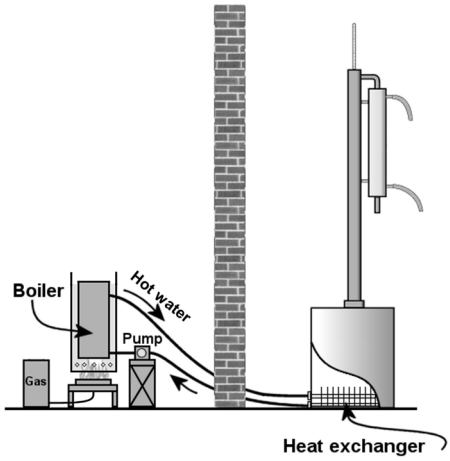


Fig. 3-9

A coil of tubing is mounted inside the boiler, in direct contact with the liquid. Hot liquid or steam flows through the coil, heating the boiler's contents. This arrangement requires a separate heat source, and is also common in industry. This is equivalent to an immersion element, but the rate of heat transfer and the highest temperature the boiler's contents are exposed to tend to be lower.

Relationship between the Boiler and the Condenser

The boiler must not produce more vapor than the condenser can handle. In a still, all the heat that's produced must also be removed. Some will be lost if the system isn't insulated well, but let's assume that we have perfect insulation, and all the heat coming from the boiler has to be removed by the condenser. This means that we must know how to match the condenser's capacity to the power of the heater element.

Let's start by assuming that we have a 1 kW heater element (1000 watts).
If you look in Appendix 1, or in an engineering handbook, you will find that 1 Watt of Power will deliver 0.2388 calories per second, 14.3 calories per minute, or 860 calories per hour.

Since 1 calorie raises the temperature of 1 gram of water 1ºC, 1 kW of heat will raise one liter of water 14.3ºC (25.7ºF) per minute. You now we have a good rule of thumb for connecting the power of the heater to the operating parameters of a "perfect" condenser:

1 kW of boiler power will raise the temperature of cooling water flowing at 1 liter per minute by 14ºC (26ºF)

As long as the cooling water is below the boiling point, the temperature doesn't matter. Cooling water entering at 20ºC will come out at 34ºC. If it enters at 100ºF, it will come out at 126ºF.

If you double the power of the heating element, you can double the flow of cooling water to keep the 14ºC temperature difference, or accept a temperature rise of 28ºC (50ºF). The choice is yours, and just a question of how you want to operate.

In practice, you should make a condenser a bit larger than needed for your heating element. If you build a small condenser, you should use a small heater element, but with a large, efficient condenser, you can consider a larger heater element.

This rule of thumb applies to a basic pot still, but more advanced still designs limit the types of condensers and the amount of heat you can use. The next section discusses these different types of and some of the choices you have to make in designing one.

The choice of boiler power also controls how fast you will be able to produce distillate. Let's examine these relationships.

It takes 540 calories of heat to convert 1 gram of water to vapor. This is the **Latent Heat of Vaporization** (LHV) of water. Each Watt of heat delivers 14.3 calories per minute, so a 1 kW heater will deliver 14,300 calories per minute or 860,000 calories per hour. Dividing these numbers by 540 calories per gram means that a 1 kW heater will produce about 1,600 grams of distilled water every hour, or about 30ml every minute, because pure water's density is 1.0 gram/ml.

The latent heat of vaporization of pure ethanol is 204 calories/gram, so the same 1kW heater will produce 860,000 / 204 = 4,216 grams of ethanol vapor per hour, or about 70 grams per minute. The density of ethanol is 0.791 gm/ml, which means it will produce 5.3 liters of ethanol per hour, or 89 ml every minute.

When distilling a solution of water and ethanol, you can expect to produce between 30 and 89 ml of distillate every minute with a 1 kW heater, depending on the mix you have in the boiler and how well your still concentrates the ethanol.

Condensers

The purpose of a condenser is to extract heat from a vapor so it condenses into a liquid. It's as simple as that. Or is it?

Supplying energy to something can be as easy as striking a match. Removing it can be much more difficult. When we discussed vaporization, we showed that it takes a lot of energy for molecules to move from the liquid to the vapor phase, and vice versa. When a molecule goes from the vapor to the liquid phase, this same energy is given up, and it must go somewhere. This is a great deal of energy, particularly in the case of water, so any method we use must be very efficient if the condenser is going to be a reasonable size.

Factors Affecting Condenser Efficiency

Surface Area and Temperature Differential

Transfer of heat is directly proportional to the amount of area available for heat to pass through, and to the difference in temperature between the coolant and the substance to be cooled. If you double the area, you double the heat transfer. If you double the difference in temperature, you double the heat transfer. The basic design of any condenser is maximize the surface area for heat transfer.

Thermal Resistance and Thickness

The type of material used to make a condenser is very important. Most handbooks detail the "thermal conductivity" of materials, but we think it's simpler and more useful to think in terms of "thermal resistance". The numbers are easier to comprehend, and thinking about how well materials **hinder** the flow of heat can lead to better insights in condenser design. Since the materials we use to make a condenser form a barrier between the coolant and the vapor we want to condense, it's helpful to know how high those barriers are.

On the scale below, copper (which is an excellent heat conductor) is given a resistance rating of 1. It's 3 times more difficult for heat to flow through brass, 490 times for glass and 13,000 times for air. In fact, air is a very good heat insulator.

Substance	Thermal Resistance
Air	13,000
Cork	7,800
Liquid ethanol	2,354
Wood (dry woods, average)	2,000
Water	716
Glass	490
Plastics (PTFE, polypropylene, etc)	200
Stainless Steel - 321	27
Stainless Steel - 410	16
Brass	3
Aluminum	2
Copper	1

This table compares these materials at equal thickness. If the thickness is halved, heat transfer will double, and if thickness is doubled, heat transfer will be cut in half.

If heat transfer were the only consideration, copper would clearly be the best material to use. So why are laboratory condensers made of glass when it has such high thermal resistance? There are three main reasons:

- glass is an inert material that can withstand the corrosive effects of most chemicals,
- skilled glassblowers can easily fashion intricate designs,
- chemists can see the results of reactions, like changes in color.

In a laboratory, these factors outweigh the high thermal resistance and inefficiency.

In the distillation of ethanol, we're not dealing with corrosive materials, we don't need intricate designs, and we don't need to see what is happening, so we can use materials like copper and brass.

The thermal resistance of the material used for a condenser is very important, but it doesn't tell the whole story. When a vapor condenses on a cool surface, it coats that surface with a thin film of liquid. Compare the thermal resistance of liquid ethanol and cork. As soon as ethanol condenses on a surface, it forms a layer of heat insulation that drastically reduces the efficiency of the condenser! It pays to ensure that the distillate runs off the cooling surface rapidly and is not held up in any way. Smooth surfaces are best because they don't hold up the condensate. In the same way, if you use fins or spines, they should be oriented in the direction that condensate will flow.

Turbulence

A vapor molecule will only give up its heat when it actually contacts a cool surface. A vapor molecule 3mm ($^1/_8$ inch) away from the cooling surface sees no cooling effect. The normal motion of the vapor molecules will ensure that contact is made eventually, but increasing the vapor's motion will increase efficiency (just like blowing on a spoon of hot soup). Anything that increases turbulence in the vapor increases efficiency, and even a slight disturbance of the vapor flow is enough. Cooling vanes often have small twists and bumps for this purpose.

This principle applies to liquids as well as gases. The more turbulent the flow of coolant, the better the heat transfer. Static (unmoving) water is a reasonably good heat insulator, which is why a wetsuit can keep you comfortably warm while swimming in cold waters.

Flow Direction

Heat exchangers are usually far more efficient if the cooling liquid and the substance being cooled flow in opposite directions. The simple explanation for this is that it keeps a larger temperature difference over more of the heat transfer surface. Many models exist to describe this behavior, and the deeper you look, the more complex they get. Fluid thermodynamics is beyond the scope of this book, but you can do the simple experiment to see how it works in your condenser.

Types of Condenser

Flat Sheet

The survival still is a classic example of a flat sheet condenser. The plastic sheet has plenty of area, but makes a poor condenser because it is not well cooled and also has high thermal resistance. Placing one end in the shade improves the performance, and providing more cooling through evaporation makes it work even better. Placing a sheet of metal on top of the plastic above the collection cup boosts the efficiency tremendously, because metal has much lower thermal resistance than plastic and "wicks" the heat. These small modifications can make the difference between too little water and enough to keep you alive!

Liebig Condenser

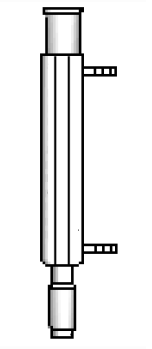
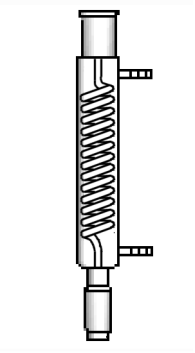


Fig. 3-10

The simple Liebig condenser is commonly used in laboratories and schools. It is just a straight tube that carries vapor through a cooling jacket of water. A length of plastic tubing surrounded by air is a Liebig condenser in principle, and you can't get simpler than that! Clearly, the longer the tube, the more cooling area, and the colder the water you pass through the jacket the better it is at condensing vapor. In principle, this condenser may be used "in reverse" by passing the coolant through the central tube and the vapor through the jacket, but its efficiency will be poor.

In use, the orientation of a condenser can have a large effect on its efficiency. As we all know, hot air rises and cool air sinks. Feeding hot vapor into the top of a condenser will be pushing it against its natural direction of movement, slowing it down and giving it more time to condense. If you feed hot vapor into the bottom of a condenser, it will tend to move rapidly to the top and require more cooling power. This is why diagrams of laboratory set-ups show the condenser sloping down at an angle, with the vapor entering at the top.

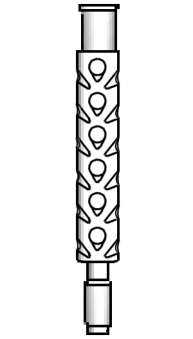
Graham Condenser



If you lengthen the central tube in a Liebig condenser and wind it into a spiral, you have created a Graham condenser. The moonshiner's "worm" in a barrel of water is a Graham condenser if you consider the barrel of water as a large water jacket. The Graham condenser must be used vertically. If it is set at an angle, liquid will settle in the bottom of each turn of the coil, and block the flow of vapor.

Fig. 3-11

Vigreux Condenser



The Vigreux condenser looks complicated but is just a Liebig condenser with its surface area increased by pushing indentations into the sides of the tube. Many variations on this theme exist. It's easily made in glass, but would be a nightmare to duplicate in metal.

The metal equivalent of a Vigreux condenser is the hedgehog, where surface area of a metal tube is increased by fitting it with fins or spines on the inside, the outside or both. This significantly increases the efficiency of a condenser.

Fig. 3-12

Hedgehog Condenser

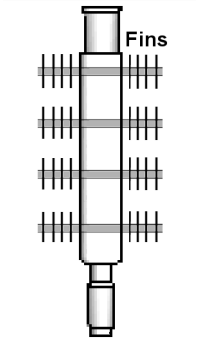


Fig. 3-13

Firebox or Shotgun condenser

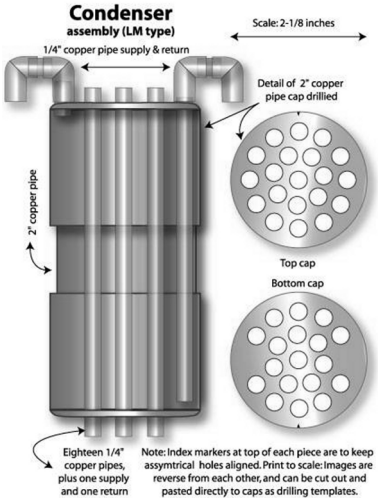
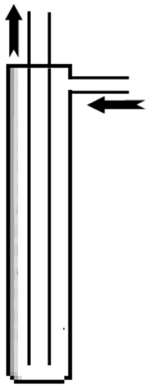


Fig. 3-14

Line a bunch of vapor tubes up side by side, and enclose them in one cooling jacket. This is called a **Firebox** or **Shotgun** condenser. The "Firebox" name comes from the fact that this same design was used in steam locomotives to generate steam. It is sometimes called a "Shotgun" because it features several parallel tubes, or "barrels".

The Cold Finger



All the designs we've considered so far carry the vapor on one side of a sheet with coolant on the other side, or inside a tube surrounded by coolant. The Cold Finger condenser reverses that, and is a tube carrying coolant that is inserted into the vapor to be condensed. This is a simple and very useful condenser that can easily be dropped into the top of a column of vapor, and is also easily removed and cleaned.

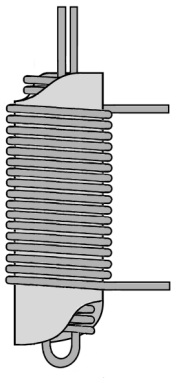
Fig. 3-15

Reflux Coil

You can increase the surface area of a Cold Finger by winding it into a coil, just like in the Graham condenser. This is the principle of a **reflux coil**. Like the Cold Finger, the reflux coil is usually inserted in the top of a column.

Fig. 3-16

The Gloved Cold Finger



The Gloved Cold Finger combines both types of condensers into one, by placing a jacket or coil around the column of vapor and inserting a Cold Finger or Reflux Coil inside it. This is an extremely efficient design, because cooling is applied to the vapor from two directions.

Fig. 3-17

Types of Still

The Pot Still

The Pot Still is the simplest possible still, consisting of a boiler (or evaporator) directly connected to a condenser.

The Survival Still and the laboratory retort both qualify as pot stills.

A more sophisticated pot still will have a boiler (usually electrically heated) with a tight-fitting lid and short tube to carry the vapor to a condenser. The alembic, with its very long spout, used air for cooling, but modern stills usually use water cooling. This simple, straightforward design makes the pot still attractive.

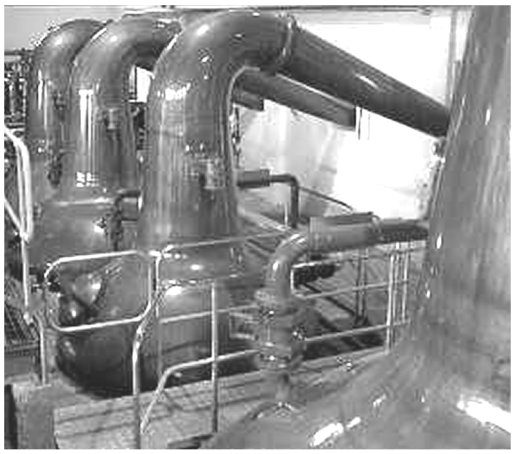
The disadvantages of a pot still are that the strength of the product can be fairly low, and it does not do a good job of separating out the congeners.

Fig, 03-18

Typically, a pot still will produce 35% ethanol from a 10% wash. Early in the run, the concentration is higher, starting at about 60%. The concentration drops steadily throughout the run as the ethanol is removed from the boiler. We discuss this effect thoroughly in Chapter 4.

You can discard the first part of a run, containing the most volatile congeners, (called **heads**), and stop the run when the less volatile ones (called **tails**), start to appear, but the middle portion of the distillate will still contain a fairly high proportion of them. These materials help produce the true flavor of whiskey, rum or brandy, but a more sophisticated still offers greater control over these trace components.

The traditional Whiskey Still



You can increase the final concentration of ethanol by re-distilling, and this is exactly what is done in practice. Two or even three distillations are done before the product is ready for storage and maturation. This traditional process works well, but is time and labor intensive. Re-designing the still can save a considerable amount of both, by creating reflux.

Whiskey stills are large copper pots topped by lovingly crafted copper alembics. Each distillery has its own unique alembic design, perfected through years of trial and error. These designs are carefully maintained and duplicated, even down to all the dents and blemishes. In the complex environment of a still, very small changes can have very large effects.

Fig. 3-19

Tradition and beauty are not the only reasons for using copper. Several distilleries have tried substituting stainless steel for copper, with bad-tasting results. Changing some of the parts back to copper restored the proper taste. Both physics **and** chemistry are involved in the process.

The alembic domes are not insulated, and are deliberately left open to the air for cooling. Vapor condenses on the inside surface to form liquid reflux, and this in turn re-evaporates, with the result that vapor becomes stronger and the reflux weaker. This process, called "fractionating", can repeat itself many times in a good whiskey still.

After making its way to the top of the alembic, the vapor flows through a special tube called a **Lyne arm** or **Swan's Neck** to the condenser. The size and shape of the Swan's Neck help establish the rate of production and the amount of turbulence in the alembic dome, controlling the amount of reflux processing that occurs. The shape of the alembic, the height of the still, and the angle of the Swan's Neck all have a marked effect on the quality of the product.

Whiskey is usually distilled twice, sometimes three times. The first distillation is performed in a large **Wash Still**, heated directly by fire or steam. The entire fermented mash (including the grains) is boiled, producing a crude and fiery distillate known as **low wines**. Boiling the mash produces many of the compounds that define the flavor and character of a whiskey. The spent mash contains a lot of protein, and is usually fed to animals.

The low wines are processed in a smaller **Spirit Still**, which is the traditional whiskey still discussed above. Three distinct fractions are collected from the Spirit Still. The **foreshots**, containing the most volatile congeners, are the first material to appear. The main part of the distilling run produces primarily ethanol and water, while the **feints** appear last and contain the fusel alcohols and other low-volatility congeners. Small portions of the foreshots and feints are sometimes added to succeeding batches of low wines to balance the flavor. The **spent lees** remaining in the still are sent to the sewer.

The Fractionating Still

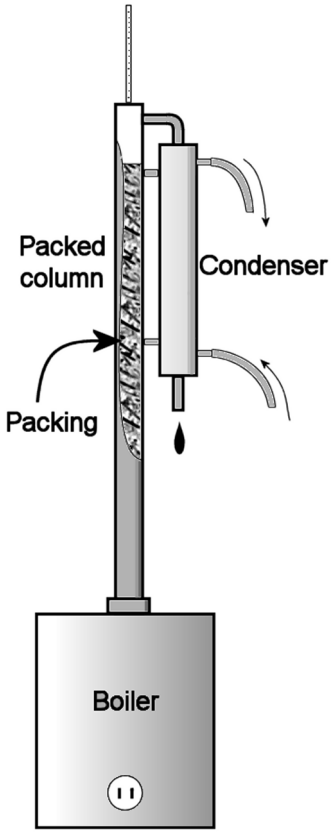


Fig. 3-20

The Fractionating Still is a compact device for increasing the amount of condensation and evaporation of reflux. A fractionating still has a vertical tube called a "**fractionating column",** filled with **packing** material, between the boiler and the final product condenser.

It's important to note that the "fractionating column" is just a **part** of the whole still, just as the boiler is a part. If this "column" were empty and had no packing material, then it wouldn't be a "fractionating" column, but would simply be a tube carrying vapor from one place to another, like the Swan's Neck in a whiskey still. The addition of packing promotes the process of "fractionating" we saw happening on the copper dome of a whiskey still. A "fractionating column" is used in both the "fractionating still" which we're about to describe, **and** the "compound still" we describe later.

The packing material in the fractionating column needs to have as much surface area as possible, and at the same time have as much open area as possible so vapor and liquid can flow freely through it. Many different kinds have been developed over the years. We discuss packing at length in Chapter 8.

The vapor rising through the column condenses on the packing, heating it up. As more vapor enters the column and condenses, the heat released re-evaporates the more volatile components of the reflux, increasing the strength of the vapor in the column and weakening the condensed reflux. The new, stronger vapor has a lower boiling point because of its higher volatile content, and will condense further up the column, starting the process over again. The depleted liquid has a higher boiling point, and will drip down the column until it finds a hotter region and evaporates again.

As this process repeats again and again, the most volatile components find their way to the top of the column, and the least volatile drip back into the boiler, and the temperature in the column is lowest at the top and highest at the bottom. The longer the column is, the more cycles of condensation and evaporation, and the better the separation of components.

In a 1 meter (just over a yard) fractionating column filled with high-efficiency packing, hundreds or even thousands of condensation and re-evaporation cycles take place. This is equivalent to re-distilling dozens of times with a pot still. The net result is a product with a very high percentage of volatiles indeed, even up to 90%. The key limitation of a fractionating column is that the vapor flow must be slow enough for the fractionating process to work efficiently. In a fractionating still, the relationship between **fractionating column capacity** and boiler energy input is critical. These factors are discussed in detail in Chapter 8.

The Compound Still

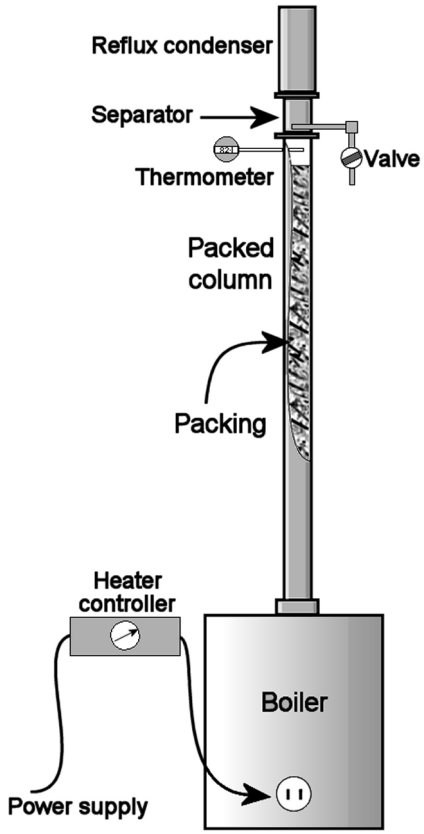


Fig. 3-21

The Fractionating Still provides a rich product, but one that is a mixture of volatile components. It does a better job than the pot still, if it is not driven too hard. The best product is obtained at the slowest operation.

The **Compound Still** offers a way to obtain almost perfect separation of each volatile component, in order of increasing boiling points. A compound still has a "reflux" condenser on top of a fractionating column, which returns most or all of the condensate to the column.

This is a natural extension of the simple fractionating still, and the addition of an additional component to the top of a fractionating column is why we call it a "compound still". The process of condensation and re-evaporation from the packing is exactly the same – except at the top of the column. In the **compound still**, the rich vapor at the top is condensed and returned, rather than being removed. When this cooled reflux is re-evaporated in the topmost zone of the column, the vapor is enriched again. This vapor is then condensed, and returned for another cycle of purification. With each cycle the product gets closer and closer to the best separation possible.

Over time, the most volatile component will find its way to the top of the column, and the less volatile components will work their way downward, establishing a state of **equilibrium**. The top zone of the column will have a constant temperature, equal to the boiling point of the most volatile component concentrated there, and it becomes a constant composition "reservoir zone". Below this zone, temperature increases as you move toward the boiler as the normal interchange between vapor and reflux **condensing in the packing** re-asserts itself. The net effect is to shorten the fractionating column as space is needed at the top for this "reservoir".

When you begin to remove some of the top condensate, rather than return it to the column, the equilibrium is disturbed. If you draw it off very slowly, the column can adjust and form a new equilibrium as the fractions lower down slowly move up the column. If you take it too quickly, equilibrium is lost and the whole device reverts to behaving like a simple fractionating still.

One way to think of this is as if the column is a high wire artist carrying a sack of potatoes. If you suddenly took a sack of potatoes from him, he would lose his balance. Take one potato at a time and you won't affect his equilibrium very much. Take potatoes at a steady rate and he will adjust to this steady routine, establishing a new equilibrium. A compound still works in precisely the same way. If a small proportion (say 10%) is removed, vapor further down the column moves up slowly, maintaining its equilibrium as it goes. If you get greedy, and take too much too quickly, the upward rush disturbs the equilibrium and contaminates the product.

The compound still introduces a major new factor to the distillation process: CONTROL.

The compound still enables you to take a mix of volatile liquids and to separate and collect the constituents one at a time in order of their volatility. The lightest fractions, such as ethyl acetate, are the first to be isolated, which means that you can remove them **entirely**. After the lightest fractions, you are then able to collect the purest ethanol achievable by distillation: the azeotropic mixture of 96% ethanol and 4% water.

The Coffey Still

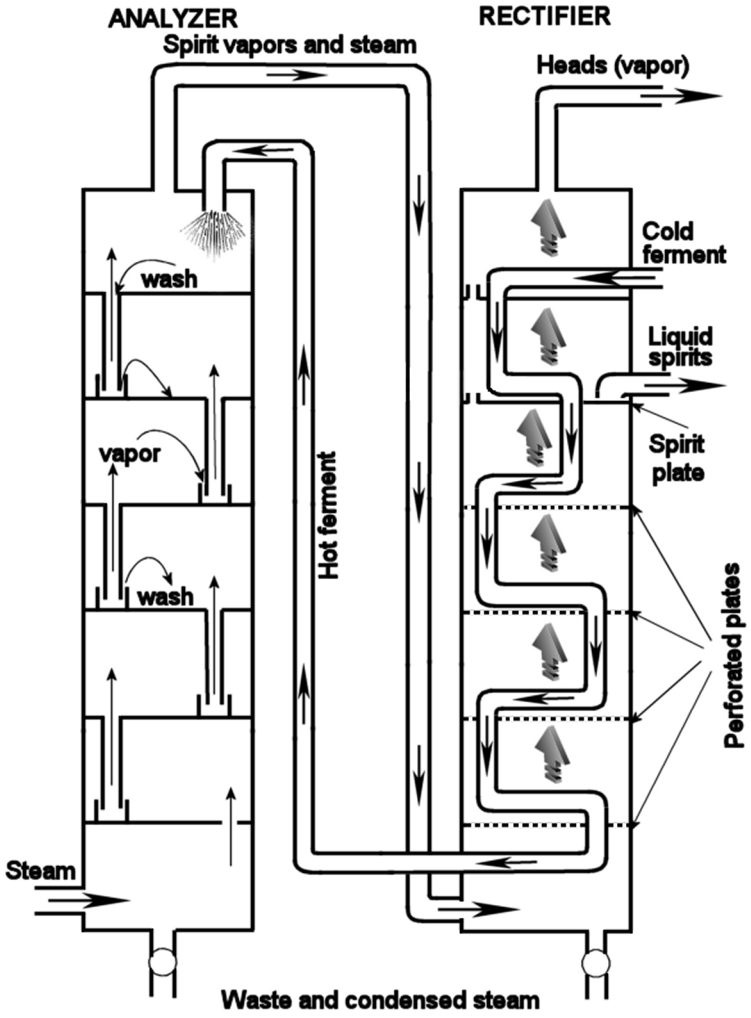


Fig. 3-22

In 1831, Josef Coffey patented a still design that revolutionized the whiskey and spirits industries. The **Coffey still**, also known as the **patent still**, was the first continuous still to achieve commercial success.

The patent still combines two batch operations into one single process. The **analyzer** performs the function of the wash still. Steam is fed into the base of the analyzer and pre-heated wash into the top. They meet on a series of perforated plates, condensing the steam and boiling the wash. Alcohol and water vapors rise to the top of the column. The spent wash runs down and is removed at the base.

The **rectifier** takes the place of the spirit still. Vapor enters the base of the rectifier and rise through chambers containing a long coil that carries conveys wash to the analyzer. Vapor condenses on the coil, warming the wash, and a large amount of reflux is generated to interact with rising vapors. Many more cycles of reflux and evaporation take place than in the traditional still. Once started, it runs continuously.

Because the action of the rectifier extracts a lot of congeners, the distillate has less flavor and aroma than traditional whiskey, and requires less time to mature. Increasing the amount of rectification removes even more congeners, giving a flavorless product used to prepare vodka or gin.

Continuous Fractionating Still

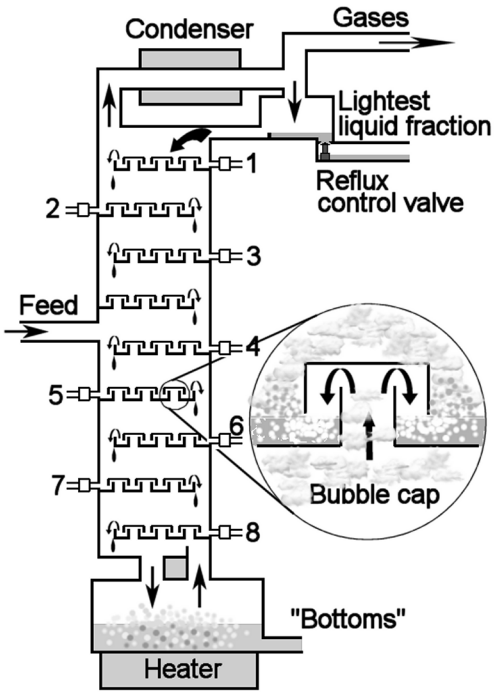


Fig. 3-23

The "recycling" principle at the top of a Compound Still applies all the way down inside a **Continuous Fractionating Still**. The Coffey still mentioned previously was an early move in this direction. Changing the rectifier section to a packed column with a reflux condenser on top creates the modern Continuous Fractionating Still used in many industries like oil refining and industrial alcohol production, better known as a "**Fractionating Column**".

In a **long** column at equilibrium, each fraction (component of a mix)will concentrate at a level corresponding to its boiling point. The temperature above this point is lower than this fraction can exist as a vapor, but the higher temperature below makes it difficult for that fraction to remain liquid. Picture it as a hot "fountain" of material that rises as far as it can as a mixture of liquid and vapor. The top of this "fountain" is behaving in the same way as the "reservoir" we made at the top of a compound still by putting a reflux condenser on top, and the constantly re-circulating material at the top of the "fountain" becomes very rich in the component that has reached the limit of how far up it can go. The column of a continuous still has many such "fountains" inside it, each representing a particular fraction. Of course, as these are not individual "fountains", but are mixed up together, none of these fractions are completely pure, because more volatile compounds are passing through on their way to the top. However, by extracting fractions at the top of their individual "fountains", we can ensure that we have the richest concentration possible.

Feeding the raw material into the middle of the column improves the quality of the separation. The upper half of the column deals only with the lightest and most volatile materials, while the lower half deals only with the heaviest and least volatile. The lighter materials do not have to pass through zones of all the heavier ones, and purer fractions are collected.

These regions are usually named for the principle component extracted there, eg. propanol, naphtha, etc. Industrial production is usually more interested in quantity than absolute purity, so the slight contamination of the fractions is not a problem. When completely pure compounds are needed, smaller quantities may be purified in other equipment.

If you are thinking that this might be a good way of running a small still, please remember the word we underlined at the beginning: "**LONG**". There just isn't the room inside a small, 1 meter long column to set up the dynamic equilibrium described above. A commercial column can be as high as a 20 floor building! The best we can do is use a small compound still and draw off the fractions one at a time from the top. Mind you, this is not really a disadvantage, for it's the fraction that reaches the top that is **really** pure!

CHAPTER 4

EQUIPMENT DESIGN

In this chapter we discuss the design and manufacture of stills.

Please remember the warning we gave at the beginning of the book:
Distillation of alcohol is illegal in some countries, and stills **intended for use** in processing of alcohol should neither be imported nor made in those countries. Furthermore, in some countries, even a still intended for purifying water or some other chemical compound must be registered if it exceeds a certain size. It is extremely important that you make yourself familiar with the laws of your area, and follow them! The consequences can be dire.

Materials

One of the easiest materials to obtain and use is copper. Copper plumbing tubing is readily available in most places in a variety of diameters and gauges, in almost any length you need. It's an easy metal to work, and parts may be easily joined with solder at low temperatures. Hardware stores stock many different types of connectors, allowing almost any kind of junction to be made. Copper is a very good heat conductor and since small gauge tubing is fairly easily formed into coils, it is the material of choice for making condensers.

More experienced metalworkers may want to use brass, at least for columns, flanges and other small components. Brass, like copper is easily worked and soldered. The advantages of brass include the facts that it's more resistant to knocks and dents and it is excellent for silver soldering, which is much stronger than the more common plumbing solder. Brass has five times the thermal resistivity of copper, making it a good choice for a column that needs to be insulated against heat loss. Also, it's beautiful when polished and doesn't tarnish as readily as copper.

Stainless steel is perfect for boilers and columns because it is very resistant to corrosion and has high thermal resistivity. On the other hand, it is notoriously difficult to machine, difficult to solder, and requires specialized welding techniques. It can be brazed and silver soldered, and off-the-shelf clamps that require no soldering or welding may be used (once the clamps are attached to the tubing and equipment, of course!)

If you want to make specialized parts, you will need appropriate workshop facilities and experience. This makes copper, with its ease of working and wide variety of ready-made parts, the starting material for most hobbyists.

Many people consider glass, because it is often used in laboratories, and for its aesthetic appeal. Borosilicate glass (Pyrex®) has excellent tolerance to corrosive materials, which is why it is used in laboratories. It has extremely low thermal conductivity and this makes it ideal for columns, but not at all good for condensers. If you do decide to use glass then you will need to find a good glassblower since it takes great skill to make anything in this material, or confine yourself to ready-made parts you can buy off the shelf.. You should also bear in mind that connecting a glass column to the boiler or to a head unit requires special clamps and relatively complicated seals … and a great deal of care! Glass is very fragile and easily broken. To be practical, glass should only be considered if you're dealing with small items, like a botanicals still, or if you can't achieve the results you want by using metal parts. In the end, it all comes down to a matter of personal preference.

Plastics should usually be avoided because many of them contain materials that can be leached out by hot vapors, and particularly by ethanol vapor. Some food grade plastics retain their strength at high temperatures and don't introduce unpleasant flavors and smells, but test them carefully to make sure that they will withstand the conditions of distillation.

Solders

Low-temperature solders, the kinds commonly used for plumbing and electrical work, are available in two basic categories – lead-containing, intended for electrical connections and mechanical work, and lead free, intended for use in plumbing of water supplies. We strongly advise using lead-free solders for **all** components of a still.

Silver solders are alloys of tin and silver (usually around 96% tin, 4% silver) and are used when a particularly strong joint is required. Unfortunately, silver solders melt at a much higher temperature than soft solders and are therefore more difficult to use.

Brazing materials are special types of brass that melt at temperatures similar to silver solder. Unlike solders, brazing materials flow slowly when melted, and can fill large voids, allowing mismatching parts to be attached to one another.

Seals

Before looking at any designs for systems or parts, we need to discuss sealing. Ethanol is a very volatile substance, and its hot vapor will escape through the tiniest gap. Every time you join one part to another, and those parts will be carrying vapor, you should ask yourself, "is it **really** 100% gas tight?" A slight water leak in a condenser is annoying, but not dangerous. However, if hot ethanol vapor can escape, it will spread everywhere. This is not only unpleasant and wasteful, it is potentially dangerous, because in a confined space it can form an explosive mixture with air! Since **safety comes first**, **always** bear in mind that ethanol and gasoline are very similar when it comes to flammability and explosive characteristics. Treat it with respect and keep it where it should be with good seals and gaskets.

Never rely on screw joints alone to provide a vapor-tight fit, even if they're the tapered kind supposed to seal themselves. Professionals always use some sort of sealing compound with threaded joints, and so should you. Get some thread seal tape, string, or jointing compound and always use it on every screwed vapor joint. These are all readily available. The best all-around choice is PTFE (Teflon ® is a well-known trade name for this) plumber's tape. It is cheap, solid, durable, and will not contribute any flavors to the vapor.

For all other joints and sealing points, using ordinary rubber for seals and gaskets is out! Hot ethanol has the ability to leach out flavors and smells from almost everything, and most types of rubber are right at the top of the list. Even the kind used in glass preserving jars is no good in a still. This type of seal works well for jams and preserves, but will ruin any batch of ethanol you produce. The best material for a seal or an O-ring gasket is Nitrile®, which is inert to ethanol. Teflon® is also suitable, although more expensive. Take care though, because Teflon® absorbs hot ethanol and can become spongy, requiring mechanical support of some kind. (This is not an issue in threaded or clamped joints, which are intrinsically self-supporting.)

A very useful alternative is silicone RTV (Room Temperature Vulcanizing) sealant. This comes in a variety of forms and is made by many manufacturers. Many of these sealants are intended for outdoor use and contain mold inhibitors. The type you want to is marked **food grade** or **suitable for aquariums**, contains no toxic substances and is clear – many of the other types are colored. Apply a very thin layer of petroleum jelly to one of the surfaces you want to be sealed (so the cured seal will release) and apply the RTV sealant to the other. Hold or clamp the surfaces together until the sealant has cured and you will have a perfect ethanol resistant seal that is permanently attached to the surface that was not coated with petroleum jelly. If you want the seal to be released by both surfaces, then smear the petroleum jelly on both of them.

If you can't find of any of these materials, there's nothing wrong with using the old plumber's trick of winding a bit of frayed string around threads to ensure a good seal, or of making simple gaskets for flat-faced flanges out of a few sheets of ordinary paper. It is a nuisance to have to replace these simple gaskets whenever you take the joints apart, but they are effective. The only time you will encounter difficulties using these simple methods is if you try to make large gaskets to seal, for example, a lid on a big pot. Here, the old moonshiners's technique was to use a flour-water paste to make seals, and this worked very well, though they could be difficult to clean up after baking in place.

Types of seal

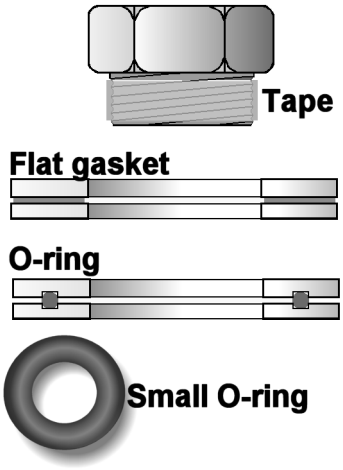


Fig. 4-1

The illustration here shows the three main types of seal you might use.

Flanges

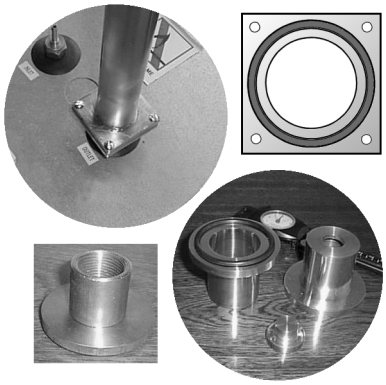


Fig. 4-2

Flanges are very useful devices for connecting two items together and are easily made if they're not readily available at your local store. A flange connection is simply a pair of flat plates attached to the two items to be joined together. The plates can be screwed or clamped to each other, with a seal of some sort between them. Flanges are sometimes a better choice than screw-in fittings because they offer great mechanical strength and can be sealed very easily. Flanges may be round or square. Round flanges are the most common type, and may be connected by either bolts or clamps. Square flanges are easily made, and are an ideal means of connecting small tubes together because the screw-in fastenings sit nicely in each corner of the square well away from the round tube. You may use flat gaskets for seals (like in a car engine) or can use O-rings, preferably sitting in matching grooves cut into each flange (you'll need a lathe to do this effectively).

We show you how to make flanges in Chapter 7.

Boilers

It's easy to worry too much about building or acquiring a boiler. There is an incredible variety of materials, designs and heating methods to choose from, but in the end, all we really want to do is gently boil some liquid and contain its vapors. Amateur distillers frequently use boilers in the range of 4 to 50 liters (1 to 12 US gallons). You should choose your size by carefully considering how much you intend to ferment, and how often you plan to run your still. It is easy and common to build something much larger than you really need or want.

You can build a boiler from a wide variety of containers, so long as they are sturdy, can be tightly sealed, and either set on a hotplate or fitted with an immersion element, jacket or coil. People have used pots and pans, stainless steel milk cans, commercial beer kegs, and even a vacuum cleaner! In general, stainless steel is an ideal material for a boiler, but any material can be used that will stand up to the temperatures and conditions of distilling without leaching any flavors or odors into the liquid or vapor. Use your imagination, but always remember that the boiler **must** be capable of safely holding boiling liquid for long periods of time.

Most of the vessels you adapt for a boiler will require some modification so you can attach a condenser or a column. The most common practice is to weld or solder a connecting device to the lid. Common connectors include threaded joints, plumbing unions, flanges, and slip fittings. Many people fasten the column or condenser directly to the lid, but this reduces future flexibility in how the still will be used.

Pot on a hotplate

We mentioned several types of boiler in chapter 3, but a simple pot on a hotplate is by far the most common in amateur distilling. The pot on a hotplate approach can range from a standard kitchen pot or pressure cooker on a kitchen stove up to large pots (eg a 55 gallon drum) set on an industrial cooker. This design can also be used to provide semi-indirect heat by placing the actual boiler inside a larger pot, filling the space between the pots with a heat-transfer liquid (Water, salt water, glycerin and propylene glycol are all popular choices), and heating the outer pot with the hotplate.

Cheap hotplates are usually controlled by thermostats, which can result in uneven, or surge, boiling. You can overcome this by using a heat diffuser. A thick cast-iron skillet, or a sheet of steel about 1cm (1/2 inch) thick should be fine. The metal plate acts as a heat reservoir to smooth out temperature swings. Should hot spots persist, adding a wire screen between the metal plate and the pot introduces a thin layer of air that will further smooth out the distribution of heat. Wire mesh is often used in laboratories to spread the heat from a flame, and the combination of metal plate and trapped air is used in good kitchen pots whose thick bottoms have concentric grooves machined in them.

Sand beds are another simple and effective way of controlling heat flow, and are often used in laboratories. They are used to provide physical support and close contact with objects that are not flat, like round-bottomed flasks. Just fill a flat-bottomed, shallow metal container with 2 to 4 cm (1 to 2 inches) of fine, dry sand, and sit the boiler into it. **CAUTION!** The sand will get extremely hot, and can cause nasty burns in an instant.

A hotplate with a heat spreader is the simplest and least expensive way of heating a boiler in a controlled manner.

Standard cooking pots will work well, but often require some extra work to make a vapor-tight seal, as described earlier in this chapter. Pressure cookers are often used, especially for simple pot stills, because they come with both a tightly sealing lid and a vapor outlet (the nozzle that the pressure control dingus sits on).

Used stainless steel milk cans and beer kegs are often available, and can be an attractive choice because they are very rugged and well sealed. Milk cans have a tightly fitting, removable lid about 18cm (7 inches) in diameter. Beer kegs have a much smaller opening, and this makes filling, emptying and cleaning more difficult.

Slightly Modified Consumer Goods

Another simple approach is to find and modify a piece of equipment that already exists. A wide variety of electric soup kettles, deep fryers and coffee urns are available, and most of them have heat capacities in the correct range. Probably the easiest item to modify for a boiler is the 14-20 liter (60-80cup) coffee urn. This usually has two concealed heating elements, one for quick heat up and the other for keeping the coffee hot. All you need to do is remove the percolator basket, improve the sealing of the lid, and replace the thermostat with a switch. The lid usually has an opening in the center that you can easily modify to attach the condenser or column.

Immersion element boilers

An immersion-type element is usually mounted through the side of a boiler and is in direct contact with the liquid. Immersion elements are available in a wide variety of shapes, sizes and power capabilities. Usually, these elements are designed to bolt to a flange or screw into a special fitting. You can purchase the appropriate fitting and have it welded or brazed onto almost any style of pot. Another type of fitting (the **bulkhead fitting**) is designed for mounting devices through sheets of metal, and if you have (or can create) a flat surface on your vessel, you can easily mount one of these.

Another route to an immersion boiler is to modify a small electric water heater. These “point of use” water heaters are readily available in sizes ranging from 20 to 60 liters (5 to 15 US gallons), with heating elements usually in the 1000 – 2000 watt range. They are well insulated, and have convenient and well-labeled inlet and outlet connections. The outlet will be on the top, and will become the exit path for vapor. The "inlet" may be on the top or the side of the cylinder. It is very important to differentiate between the "inlet" and "outlet" fittings, because the inlet is often connected to a tube inside the unit that delivers cold water to the bottom of the tank. It is therefore always immersed in liquid, and cannot deliver any vapor! The inlet needs to have a valve or cap attached that remains closed except when filling or draining the unit.

Water heaters have built-in thermostats that prevent them from boiling. Since you **want** the contents to boil, you must remove this thermostat from the circuit. Some water heaters have a **sacrificial anode** of magnesium or magnesium alloy, which should be removed if possible. If you are not completely sure of your skills as a plumber or an electrician, get someone to do the work for you! Many home brewers have found that a few bottles of their product can be traded for a lot of work on equipment.

Indirectly heated boilers

There are many advantages to an indirectly heated boiler, but other than the “pot in a pot” technique, they are difficult and expensive to build. You might be able to find a jacketed cooking vessel at a used restaurant supply store, and that would be the best way to create a true indirect heating system. Vessels with heating coils are generally too large to consider for a small-scale boiler.

This method is used extensively in industry, because a single boiler can heat many pieces of equipment. Industrial applications usually use steam as the heat transfer medium, because it is very efficient. Steam can be heated to very high temperatures and its own pressure will move it around the system, while hot water systems require a separate pump.

Whatever the advantages, steam heating should **NEVER** be attempted by the amateur! **STEAM IS DANGEROUS.** Because of the dangers of steam, pressurized boilers require licensing and regular inspections, and still manage to explode and kill people on a regular basis.

If you want to try this technique - and it can offer significant safety advantages if you are heating with a flame - then use hot water for the heat transfer medium. **NEVER** operate a pressurized system.

Boilers - Summary

All of these methods of heating are suitable, and each has advantages and disadvantages, so none of them can be declared the winner. Choose your design based upon your own unique situation.

You can make a boiler yourself (paying due attention to grounding and electrical safety, or to fire and ventilation if using open flame heating) or you may choose to buy a boiler ready-made. Small electrical water heaters are a good option because they're compact, sturdily made, and are usually well heat insulated (which saves on your electricity bill). Using a water heater for a boiler is described in more detail later, in the section on Pot Stills.

Boiler control

Directly heated boilers can suffer from the phenomenon of surge boiling. The more advanced fractionating and compound stills require a well-regulated and moderate heat input to operate correctly. Whatever type of boiler you have, and whatever kind of still you use it in, you do need to think about controlling the boiler.

1 kW of power will vaporize 1.6 kg of water each hour at its boiling point. That same kilowatt will vaporize 4.2 kg an hour of pure ethanol, and a mixture of water and ethanol will vaporize at a rate in between the pure products. Since fermented mixtures contain at most 20% ethanol, the quantity of liquid converted to vapor will be close to that of water, even though it boils at a temperature below 100º C. If you are re-distilling a product high in ethanol content, the rate of vaporization will be much higher.

Despite this considerable difference in weight, the **volume** of vapor produced each hour will be the same no matter what the proportions of the mix may be (chapter 2). For mixtures that are primarily water and ethanol, 45 liters of vapor are produced every minute per kilowatt of heat applied. A 25-50mm (1-2inch) diameter tube can easily handle several times this amount of vapor.

When you place packing in a column, as required by both fractionating and compound stills, the vapor path is restricted, and you have to start thinking about boiler control. Experience shows that a loosely packed 50 mm diameter column performs well with a 1 kW boiler, but is operating close to the limits of its reflux capability. 36mm (1.5 inch) is also a common column size, which reduces the power requirement to 750 Watts.

The most common control problem is how to heat a boiler full of liquid up to its boiling point rapidly, then scale back to the proper controlled simmer. A 750 Watt element would take several hours to heat up 20 liters of liquid.

The simplest method is to fit two heater elements in the boiler, one rated at 750 or 1000 watts for the voltage you'll be using and the other of any size you need to help heat up quickly. Control then simply becomes a matter of switching the larger element off when the pot begins to boil. Be careful not to exceed the capacity of the supply circuit, though, or you'll blow a fuse. You might have to use two separate circuits for the two elements.

It is possible to provide further control by changing the way in which the heater elements are wired together, and this can give you up to 4 discreet power levels. Full details of how to do this are in Chapter 7.

It is also possible to alter the output of an element by lowering the voltage. Specifically, running a 240v element on 120v will reduce its power to a quarter of its rated value. A 4000W 240v element will provide 1000W of heat running at 120v. We do not recommend this method, because it could create confusion about what devices may safely be plugged in to which receptacles.

120v power, common only in North America, is not designed for heavy power draws. The standard 120v household receptacle is rated for 15 amperes, and the largest heating element that can be legally connected to a 120v circuit is 1500W.

Variable Control

The methods we have just discussed allow power to be changed between a few pre-selected values. For most small stills, this will be more than satisfactory, and is inexpensive. If you intend to process a wide variety of different materials, or have batch sizes that vary a great deal, you might need **fully variable control**, which allows you to continuously adjust the amount of heat produced.

Most hotplates and electric stoves, have a good power control built in. This makes boiler control easy, especially if you are using a heat diffuser.

Heat control is also relatively easy with indirect heating methods, particularly the water jacket. In this case, you are concerned with the temperature of the circulating fluid, not the heater element itself. The large volume of circulating fluid smooths out the fluctuations from the heater.

Most consumer equipment (electric soup kettles, frying pans, etc.) has adjustable thermostatic control, which will not be satisfactory for a boiler, but can work as a source of indirect heat. If you are using this kind of equipment for a direct boiler, you need to lock the thermostat on high and use another means of controlling the heat output.

Methods of control that work well for isolated or concealed elements may produce problems with an immersed one, because there is no heat buffer between the element and the boiling liquid. It's surprisingly difficult to evenly control the output of a simple electrical heater element. Many ingenious methods have been tried, but most of them cannot be recommended because they can interfere with the power supply system by inducing spurious direct currents into the "neutral" line. These currents can, and do, cause major power supply fluctuations that can lead to area blackouts and expensive repairs. Recent (January 1, 2001) international regulations are quite explicit about this this and related radio frequency interference*.

These regulations have become necessary because of the cumulative effect of all the ordinary pieces of electrical equipment that people use daily – light dimmers, fan speed controls, heater controls, etc. The sheer quantity of such equipment is now so large that what used to be a minor problem has grown to major proportions.

It is important that any heater control you use doesn't contribute to this, because one boiler on its own can have as large an effect as 100 other devices. Power companies are now actively searching for large sources of interference – and a poorly controlled boiler would be a large source. **DO NOT attempt to control a large power heater element with an ordinary light dimmer, no matter what its rating.** Dimmers rely on phase switching and not only introduce direct currents into the neutral line, they also introduce multiple harmonics of the basic power supply frequency. Both these effects can damage the power supply system. Even switching a heavy load on and off rapidly without sophisticated phase control can cause damage. For this reason, don't use the old method of using a diode to halve the power delivered to the boiler. It **will** interfere with the power supply and the power company will come looking for you.

You can find the design of a control circuit in Appendix 5. This circuit is designed to be fully compliant with current regulations.

We must stress that **only** people who have experience with electrical circuits should attempt to build any of the devices mentioned in this section, because large currents are involved. If you have any doubts at all about your skills in this area, find a friend who has sound electrical experience and get him or her to make it for you.

*** International Electrotechnical Commission (IEC) regulations**:

IEC/EN61000-3-2	phase control of power
IEC/EN61000-3-3	creation of rapid voltage changes
CISPR 14-1/EN55014	radio interference relating to household appliances

Condensers

In Chapter 3, we discussed five important factors of condenser design. They are:

a. The area of the cooling surface. If you double the area, you double the heat transfer.
b. The thermal resistivity of the condenser material. Again, double this factor and halve the heat transfer.
c. The thickness of the material separating the vapor from the coolant. If you double the thickness, you halve the heat transfer.
d. Turbulence – the movement of both vapor and cooling medium relative to the cooling surface that separates them.
e. Usually, you get the best efficiency when vapor and cooling medium run in opposite directions.

We also arrived at a Rule of Thumb:

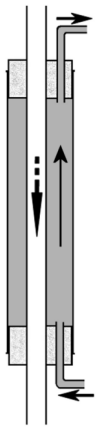
1 kW of boiler power will raise the temperature of cooling water flowing at 1 liter per minute by 14°C (26°F)

The molecules of a gas are much further apart than in a liquid, making heat transfer less efficient. It is possible to pass vapor through a tube or system of tubes and to cool those tubes with air, but it will be difficult because both materials are gases, and have poor heat transfer characteristics. The design of such a system will have to be quite clever. Car radiators work quite efficiently because the medium being cooled is a liquid and is in intimate contact with the finned radiator tubing. And on that subject, if you are contemplating using a car radiator as a condenser for your vapor - **DON'T**! Car radiators are generally manufactured with lead solder, and if they have been used, are probably contaminated with lead from the fuel. You don't want lead to get into your product, because you will be consuming it. Lead is a very nasty and cumulative poison that builds up in the body.

These factors should be considered when you choose what type and size of condenser you want to make. Since your requirements may be unique, and different from anyone else's, any dimensions that we give are only indicative. Feel free to change them slightly, as your purposes or materials require. This is the Ideas Department, and the most important ideas are your own!

We discussed the various types of condenser in Chapter 3. Now - how can you make them yourself?

The Liebig Condenser

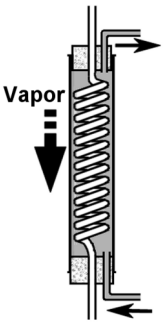


The familiar Liebig condenser has limited surface area, but very easy to make. You can increase the condenser's surface area by making it longer, or using finned tubing. Vapor is usually passed through the central tube because this contains the vapor in the smallest volume and surrounds it with cooling liquid. The Liebig condenser works most efficiently when held at an angle with vapor flowing **downwards**.

You can easily make a simple and effective Liebig condenser by holding a central tube inside a larger tube with corks. Two small holes need to be drilled through the corks or the outer tube itself for the cooling water inlet and outlet tubes.

Fig. 4-3

The Graham Condenser



Twist a small central tube into a coil, and you have made a Graham condenser, which is more efficient than the Liebig, but also more difficult to make. The Graham condenser **must** be held vertically with the vapor flowing downwards, so the condensed liquid will not collect in the coils and impede vapor flow. We believe that a finned Liebig is at least as efficient as a Graham condenser.

You can make a Graham condenser by the same simple method used for the Liebig if you leave the ends of the coil straight and pass them through corks at each end of the cooling tube.

Fig. 4-4

The "Firebox" Condenser

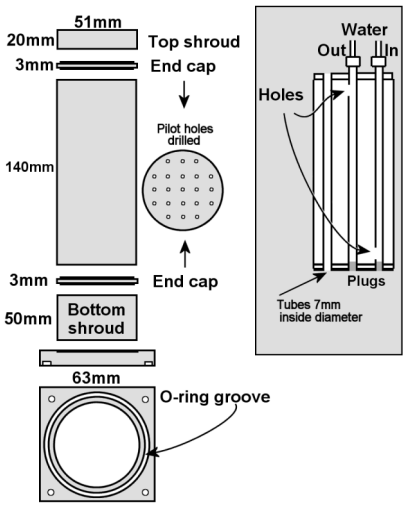


Figure 4-5

This design is useful when you have limited space, especially when the vapor is going **up** the tubes and the distillate coming **down** – the situation with a condenser on top of a column. A limitation of this design is that the tubes have to be at least 7mm (¼ inch) internal diameter or you may experience **choking**, where the surface tension of the distillate causes the liquid to block the tubes. This is the same effect that causes liquid to remain in the bottom portion of a drinking straw.

Although construction of either variation is quite straightforward, you need to be very accurate when drilling all the holes in the end plates that hold the tubes in place. This is not recommended for your first project!

The Cold Finger

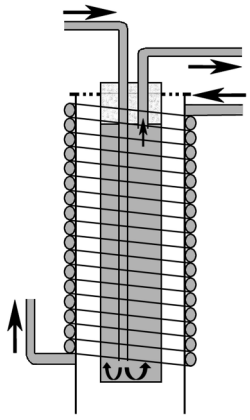


Fig. 4-6

This design is very easy to make, and useful when you want to insert a condenser in the top of an open tube. Its efficiency is poor due to the limited surface area, but may be improved by confining the vapor with a shroud keeping it close to the cold surface. It is most useful for small amounts of vapor, as in the processing of botanicals.

This design is simply made by sealing off the bottom of the outer tube (which is the "Cold Finger") and then inserting a long, open tube through the middle of a cork. A second short tube through the cork completes the water path. If you use a Cold Finger for a reflux condenser on top of a column, You can increase the efficiency by winding a copper tube around the shroud and soldering it in place. This is the “Gloved Cold Finger” mentioned in Chapter 3. This additional tube can be connected in series with the Cold Finger so the same water connection feeds both. If you do this, let the water flow through the Cold Finger first and then through the outside coil. This creates the greatest temperature difference at the part with the smallest surface area - the central Cold finger.

The Reflux Coil

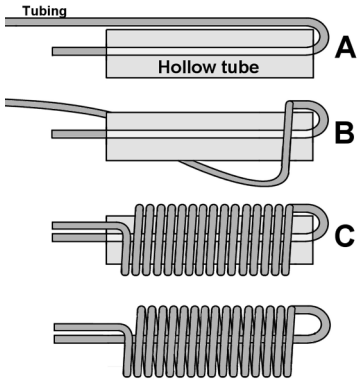


Fig. 4-7

Many people prefer to make this type of coil condenser to condense vapor at the top of a column because it is simple to make, despite its complicated appearance. Since it is most often used to condense reflux, it is simply called a **Reflux Coil**.

Soft copper tubing 5mm ($^3/_{16}$ inch) to 6.5mm (¼ inch) in diameter is the best material to use. This size tubing is widely sold in coils for use in hydraulic lines, refrigeration and some plumbing applications. The smaller diameter tubing is better, because it is easier to bend and less likely to flatten, but the larger size can be used if you're careful.

You need a hollow former to make a reflux condenser. This could be a short length of pipe with an outside diameter matching the inside diameter of the coil you want to wind. Even with 5mm ($^3/_{16}$ inch) tubing, don't attempt to wind a coil smaller than 36mm (1½ inches) inside diameter, because the tubing **will** start to flatten out. You can calculate how much copper tubing you need, but as an example, 4¼ meters (14 feet) of 5mm ($^3/_{16}$ inch) tubing on a 36 mm (1 ½ inch) former will make 27 turns, leaving 150 mm (6 inches) free at each end for the connections at the top. The length of the coiled part is just over 250 mm (10 inches), and it will fit neatly into a 50 mm (2 inch) diameter column.

Start by threading the tubing through the middle of the former, leaving about 150 mm (6 inches) sticking out. This will be where the water hose is attached. Then, carefully form a looping bend and start winding the rest of the tubing around the former, back toward the end left sticking out. If you manage to keep all the coils close together, you should end up with about the same amount left over.

After removing the former, carefully stretch the coil out a little bit. You'll find that the coils will then be spaced apart by up to the diameter of the coil tubing. Finally, bend the two feed pipes at right angles to the coil (being careful not to kink the tubing) and you'll have a convenient "hanger" to hold the coil in place when you insert it into the top of the column. With a close fitting shroud, this condenser can easily handle up to 2 kW. The closeness of the shroud encourages vapor to flow between the coils rather than past them. Loosely packing metal scrubber material around the top of the reflux coil will encourage intimate contact between the vapor and the condenser coils (this trick also works with a Cold finger).

Condensers - Summary

We have discussed the designs that we've found most useful for small scale distilling. You may think of others, and we encourage you to do so, because innovation and experimentation are the most enjoyable parts of this hobby. Throughout history, thousands of people have worked to design a better mousetrap. You can join this happy band of inventors and design a better condenser!

As you design your equipment, you may even start with the old moonshiner's coiled tube (or "worm") in a barrel of water - essentially, a Graham condenser with a very large shroud. It is efficient, and is used to this day in many commercial whiskey distilleries.

Designing and building stills

The Simplest Still

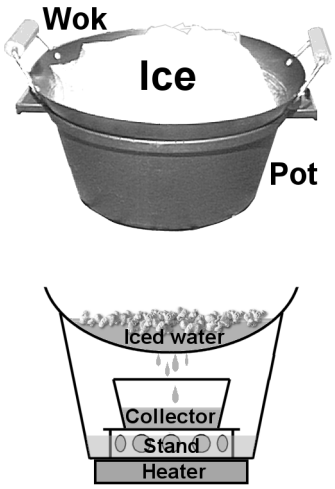


Fig. 4-8

Perhaps the simplest still you can build is the "Wok-in-Pot" device. This incredibly simple still is actually quite efficient, and may be assembled or dismantled in seconds. To assemble one, place a trivet in the bottom of a large pot, and then partially fill the pot with the liquid to be distilled. Put a smaller container on the trivet in the center of the pot, then place a wok on top of the pot and fill it with ice. When the liquid in the large pot vaporizes, it condenses on the cold wok, runs down to the bottom and drips into the smaller container. The disadvantage of this design is that you have to open it to collect the distillate, and you cannot see how it is working. It can process quite a bit of material in a series of short, small batches.

The Pot Still

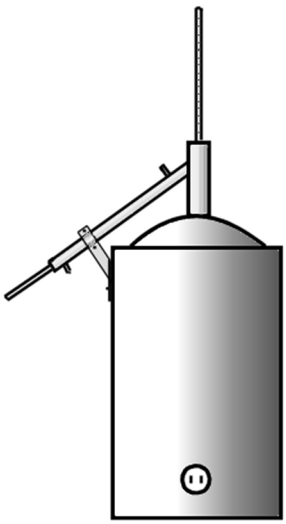


Fig. 4-9

The pot still is also a very simple device, consisting of a boiler, a condenser, and a connecting tube. Many successful pot stills have been made from everyday kitchen pots and pans. Pressure cookers are a common choice because they are sturdy, and can also provide steam for processing botanicals by steam distillation.

A common pressure cooker is more than ample for producing steam for botanicals, but rather small for producing spirits from a batch of fermented brew. Amateur distillers frequently use boilers in the range of 4 to 50 liters (1 to 12 US gallons). You should choose your size by carefully considering how much you intend to ferment, and how often you plan to run your still. It is easy and common to build something much larger than you really need or want.

Once the boiler is built, you need to connect a short tube to the top to carry the vapor to the condenser. This is usually easy with a pressure cooker, because most of them have a nozzle on top for the pressure regulator. A short length of polyethylene tubing can connect this nozzle to a similar diameter metal tube (keeping plastic parts to a minimum). This metal tube is then connected in a similar way to a simple condenser like a Liebig. If you have a modified water heater, then the tube can be screwed directly into the outlet fitting. If you have custom-built a boiler, you will have to choose how to fit the tube to the pot, and many methods are detailed throughout this book.

The pot still is an attractive option for beginners because of its simplicity, and the speed of obtaining results. Much of the equipment you make or acquire to build a pot still will be useful later on, when making more advanced fractionating or compound stills. The boilers are exactly the same, but you need to add external heater control to use the more sophisticated designs.

Warning! An unmodified pressure cooker is really NOT suitable for fractionating or compound stills, because of the tiny nozzle. If you want to use one, you should replace the nozzle with a larger fitting, which usually ruins it as a pressure cooker!

The Fractionating Still

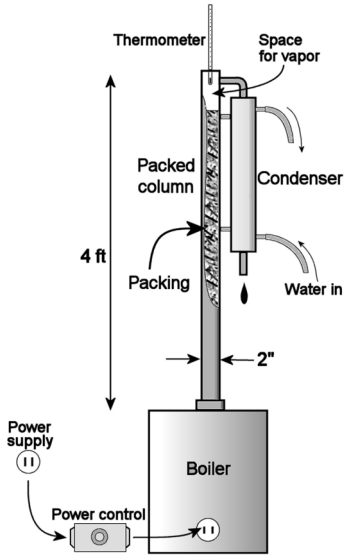


Fig. 4-10

Making a boiler for a fractionating still is exactly the same as for a pot still. However, the size and control of the heating element becomes very important. A fractionating column works because vapor repeatedly condenses and re-evaporates on the packing, constantly increasing the percentage of volatile compounds. The faster hot vapor flows into the bottom of a reflux column, the more difficult it is for reflux to condense, and the column loses efficiency. In the extreme case, you end up with a column full of hot, fast moving vapor and no reflux - a pot still.

There is considerable leeway in the operation of a fractionating column, but remember that the more slowly the vapor rises up the column, the more efficient the fractionating process will be. We have found that it works best to limit the heater element to no more than 1 kW for a 50mm (2 inch) column.

A fractionating still is only a little more difficult to make than a pot still. In fact, the only difference is the use of a packed column instead of an open tube between the boiler and the condenser. In countries where amateur distilling is legal, many manufacturers offer “reflux” stills. Should you choose to buy one, first make absolutely sure that it really **is** a fractionating still. Many of them do not have adequate surface area in the column to enable efficient fractionation, either because the column is too short, or because the packing is inadequate, or both. They are really just pot stills, and the advertising is a case of mutton dressed as lamb.

Making the fractionating column

In Chapter 3, we showed that reflux is the key to effective separation of the components of a mixture by distillation. The composition of the vapor gradually changes as it rises up a reflux column, becoming richer and richer in the most volatile components until, at the top, the vapor is very rich in the most volatile components. You can increase this separation by lengthening the column, but you will quickly reach the point of diminishing returns, because the increase in concentration grows smaller with each cycle of condensation and evaporation. The vapor and dew lines get closer and closer together toward the bottom of the curves.

A very good amateur column can be made from 1 meter (39 inches) of 50mm (2 inch) tubing. Increasing the length of the column will increase its fractionating efficiency, but increasing the diameter runs the risk of the liquid **channeling** as it works its way downward. Experiments have shown that the greater the diameter of the column, the more likely it will be that the reflux will establish "preferred paths" and run down quickly instead of slowly dripping from level to level. If that happens, the purpose of the column is defeated and the still is once again just a pot still.

Large industrial columns use physical spreader trays to hold and distribute the liquid in the column, and it seems reasonable that a similar technique would work on a smaller scale. However, we've never found any reason to explore that because a 5 cm (2 inch) column is simple to pack and provides more than enough output. Please feel free to do a bit of research!

The column should be loosely packed with packing material, leaving a gap of around 6 inches at the top and 2 inches at the bottom. These gaps allow you to insert a thermometer and other items you might want later. As we have said before, packing with marbles is a waste of time and effort. Chapter 8 tells why in detail. Commercial packing material, like Raschig rings will do very well, but with a packing made of small pieces you need a way to retain them in the column. Wire screening pushed into the end of the tube is a simple answer. Just make sure that the wire is not so fine that it impedes vapor flow. We prefer to use metal scrubber material for all the packing. It provides the best surface/volume ratio and holds itself in place in the tubing. It also gives you the choice of using copper as both packing material and chemical catalyst (again, see Chapter 8 for details).

You can use whatever means of connection best suits you at either end of the column. If you use a commercial water heater as a boiler then a simple threaded adapter will probably be best to connect the bottom of the column to the boiler. Don't worry about the small diameter of the coupling. For the rate at which you'll be operating, a 13 mm (1/2 inch) opening is quite capable of delivering all the vapor produced.

A tube leads from the top of the column to the condenser. The condenser can be as simple or as complicated as you like, but an ordinary straight Liebig condenser will do just fine. We suggest building one about 60 cm (2 feet) long, because the extra surface area will allow good cooling with low water flow. A tee junction should be on top of the column, so that a thermometer can be inserted. A simple cork will do a fine job of holding the thermometer in place, or you may wish to use a metal compression fitting as described in Chapter 7.

Finally, resist the temptation to put "cooling tubes" through the middle of the column. Those that advocate them believe that this increases the amount of reflux and so increases the efficiency of the "reflux" column. In Chapter 8 we will show that all these cooling tubes do is interrupt the thermal gradient in the column and consequently **reduce** the overall efficiency. The only place that **induced** reflux may be used profitably is at the **top** of the column, and this is the key feature of a compound still.

The Compound Still

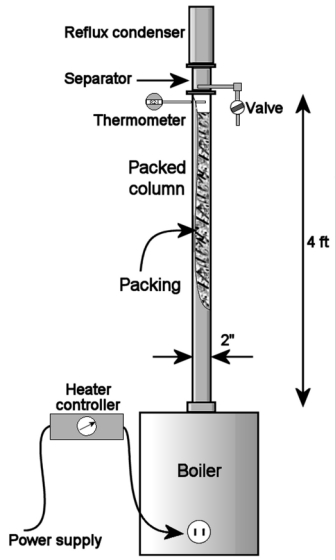


Fig. 4-11

The compound still allows you to increase the number of cycles as much as you want **without** lengthening the column. Placing a condenser on the top of the fractionating column and refluxing all the condensate creates a **closed loop**, with the reflux condensing and re-evaporating continuously in the upper section of the column. The last little bit of separation, which is the hardest to achieve, takes place in this re-circulation zone. The distillation process can now produce the most volatile component of a mixture, pure and without contamination!

Construction and operation of a compound still require a clear understanding of the processes occurring in the fractionating column. At a reflux ratio of 100%, the flow of vaporized material going upwards is matched by an equal flow of reflux flowing downwards. As the most volatile component builds up at the top of the column, the less volatile ones migrate downward to areas of higher temperature, and the least volatile ones constantly drip back into the boiler. Molecules of the most volatile component are constantly moving into and out of the reservoir zone at the top, but less volatile components cannot make it to the top. This is the state of **equilibrium**, or dynamic balance.

When you begin to withdraw product from the top of the column, it is replaced by material from lower in the column. If the product is removed slowly enough, the equilibrium can maintain itself, and the size of the reservoir zone remains the same as long as more of the product is entering the column from the boiler. If you remove product too rapidly, the impure material will move up the column faster than the column can purify it, and the reservoir zone will shrink and become contaminated.

A well-designed column can consistently produce a very pure product at reflux ratios around 90%. Even taking only 10% of the condensate causes the reservoir zone to shrink slightly, but not enough to effect the purity at the very top, where the product is being condensed and collected.

Equilibrium requires a delicate balance between the rate of vapor production, the rate of condensation, and the capacity of the column for liquid and vapor flow. Like with the fractionating still, you can apply all the power you want to the boiler until it begins to boil, but then the power has to be adjusted to a gentle simmer, to provide an even flow of vapor.

As an example, 750 watts of heat will drive vapor up a 50 mm (2 inch) diameter column at a speed of 28 cm (11 inches) per second. For a column only 1meter long (around 1 yard), that's quite rapid. Without any interference, the vapor will pass all the way through the column in only 5 seconds! In a compound still, all the vapor has to be condensed and re-evaporated many times on the packing within the column as well as being recycled time an again at the top, making the column a very busy place. Control of the heat entering the boiler, and thus of the vapor entering a compound still is essential.

The compound still head

Unfortunately, you can't make a compound still by just setting a reflux condenser on top of a fractionating column. It's a little more complicated than that. The purpose of the reflux condenser is to condense the vapor reaching it so it can be returned to the column. The job of the **still head** is to control the percentage of the distillate that is collected rather than returned as reflux.

Once again, just about any design of boiler will work well, as long as it is properly controlled. Your choice of condenser for the top of the column is affected by the fact that the vapor and the distillate will be going in opposite directions. A Reflux Condenser is probably the simplest choice, though a Firebox type will work well also, as long as the cooling tubes are large enough to avoid choking with liquid. A simple Liebig condenser will also do the job, but is quite a bit longer, making the complete still very tall.

Compound still management

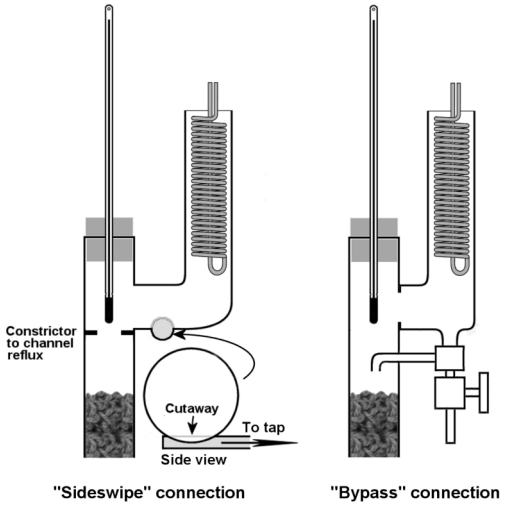
Choosing the type of condenser is simple compared to the question of how to control reflux and withdraw the product. There are three fundamental methods of accomplishing this:

- **Liquid management**. Divide and distribute the liquid condensate directly.
- **Cooling management**. Divide the vapor into two flows by managing the amount of cooling in the reflux condenser.
- **Vapor management**. Divide the vapor into two flows mechanically.

Liquid management

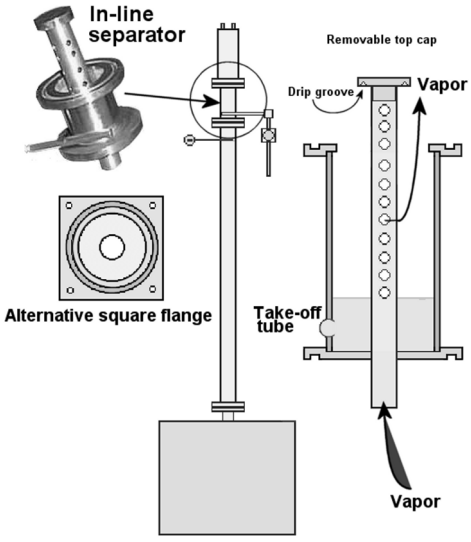
The still head does two things: taps into the reflux stream and controls how much is withdrawn. Neither the pot nor the fractionating still has this problem, because all the vapor arriving at the top is removed. A compound still is able to produce excellent separation because only a small amount of the vapor is collected. Large commercial operations solve this problem by collecting all reflux from the condenser in a tank, and running two separately controlled lines from it, a small percentage to the collection tank and the remainder back to the column.

A header tank is impractical for a small still, so the reflux dripping from the condenser is sampled directly and product withdrawn through a valve that can control very small volumes of liquid. Getting this process set up is touchy. You must have a good understanding of the rate at which reflux is being produced, and a precision (needle-type) valve for controlling the withdrawal rate.



The simplest way of sampling the liquid is to offset the condenser, with an elbow. The vapor flows upward through the elbow, and the reflux runs back down it past a sampling point. There are two ways to construct the sampling point, and both are shown in the diagram. In one, a tube is set into the bottom of the delivery/return elbow, and all the reflux from the condenser flows over the opening of the sampling tube. The other provides a separate return path for the reflux and samples from that. Both methods work well and the choice is entirely up to you. With either method, the reflux returned to the column has to drip onto the packing, rather than trickle quickly down the walls, so the re-entry point for reflux must be designed with care.

Fig. 4-12
Offset liquid management techniques)



A more sophisticated method is to dispense with bypass tubes, as shown in this diagram. Vapor flows through the holes in the central tube and is condensed. Reflux is prevented from passing back down the central tube by the top cap and collects in a small reservoir. The lowest holes in the central tube control the maximum depth of liquid in the reservoir. Product is removed from this reservoir by a sampling tube equipped with a valve to control the rate of flow.

There are many ways to sample the liquid reflux from the condenser and control the extraction rate, but these two are the most widely used. You may be able to think up other, smarter ways of doing it, and we would encourage you to do so. The biggest problems you will have to solve are the accurate control of very low flow rates of liquid reflux, and the return of reflux to the center of the column.

Fig. 4-13

Unfortunately, all these liquid management methods share the disadvantage that if the vapor delivery rate changes, the reflux ratio changes, because the valve controls only the amount of material **removed**. This is not usually a major problem with a well-controlled boiler, but can cause difficulty should the **composition** of the vapor change, as it does towards the end of a run. The temperature of the product can also be quite high, and since hot, concentrated ethanol has many similarities to hot gasoline, you should treat it with caution! All of these difficulties: the delivery rate, the composition, and with the temperature of the product, are neatly overcome by the other two types of still head.

Cooling management

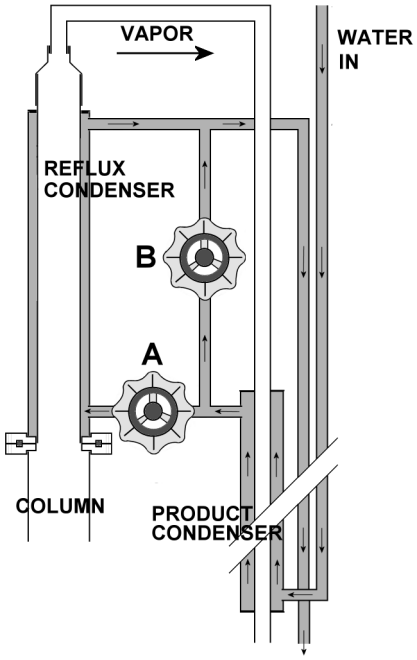


Fig. 4-14

If you reduce the amount of cooling water fed to a reflux condenser, at some point it can't condense all the vapor, and some will begin to flow past the condenser. If you do this by design, then the vapor getting past the reflux condenser can be condensed by a second condenser and collected as product.

Consider the operation of the unit in diagram 4-14. Initially, tap A is wide open and tap B fully closed. All the cooling water flows through the reflux condenser, and 100% reflux occurs. After the column has achieved equilibrium, tap B is gradually opened. As more water flows through tap B, less water flows through the reflux condenser, and vapor begins to pass by without being condensed and returned to the column as reflux. This vapor reaches the product condenser, where it is condensed as product (at whatever temperature you choose!)

We've found that turning just tap B offers more than enough control to reduce the reflux ratio to around 90%, but different stills may have different characteristics. The advantage of having two taps is that control of the water flowing through condenser 1 can be reduced still further after tap B is wide open. Closing tap A fully when tap B is wide open results in zero reflux, converting the system to a fractionating still or, if the heat input is increased, to a pot still.

This option requires constant pressure cooling water if the reflux ratio is to be kept constant for any tap setting. You can get this by using a small water pressure regulator or by making a small header tank. Also, the piping for the cooling water has to be laid out carefully to prevent airlocks from forming.

Vapor Management

Let's see how we might do by splitting the vapor flow into two parts, and then condense them separately. This also requires two condensers, but simplifies the control because the volume of vapor is much larger than the condensed liquid reflux. You don't need a tiny, precision valve, and can take advantage of the fact that vapor will divide itself up in direct proportion to the area of available outlets. If one aperture has 9 times the area of the other, then about 90% of the vapor will flow through it to the reflux condenser, leaving about 10% to flow to the product condenser. The geometry of the pipes, the viscosity of the vapor, etc will all affect this ratio, but in the system we're designing, these effects are small in relation to the large quantities of vapor, and the errors are insignificant for practical purposes.

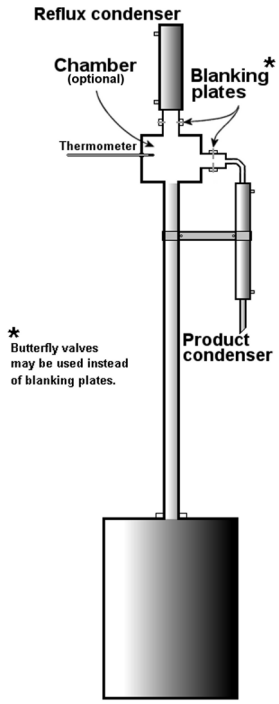


Fig. 4-15

The simplest way to do this is by inserting a pipe in the side of the column head that has exactly 1/9 the cross sectional area of the column itself (assuming you want 90% reflux). This means that the diameter of side pipe must be 1/3 the diameter of column. All that you need to control the flow of vapor into this pipe is a simple valve that is either wide open or closed shut. Alternatively, you could use a larger diameter side arm and have **complete** control of the vapor passing through it by using a **gate valve**. Gate valves are cheap, commonly used for water supplies, and they provide a straight path for the vapor to flow through.

The big advantages of vapor management are:

- Excellent control may be achieved with a cheap gate valve, large movements producing finely graduated results.
- The reflux ratio is totally unaffected by variations in the cooling water supply
- The temperature of the water in the reflux condenser doesn't increase as the reflux ratio is decreased (as it does with cooling management).
- You have control over the temperature of the final product, which is delivered at the temperature of the cooling water initial supply.

There is one disadvantage: changes made to the tap setting take around ten seconds to be observed at the collection point as collected vapor takes about this time to traverse the product condenser. Anyone who has steered a large boat will find this a familiar situation!

A design for a variable reflux Vapor Management head unit using a gate valve is at Appendix 6.

Control at the end of a run

It was noted earlier that towards the end of the ethanol run, the proportion of water in the vapor increases. The result is that as more water is carried in the vapor, the volume of distillate condensing in the product condenser will be drastically reduced. Constant volume liquid sampling with Liquid Management will result in a rapidly **decreasing** reflux ratio, just at the time you want it to remain constant, or even be increased! Cooling Management maintains a constant reflux ratio if the flow of cooling water is constant, no matter what the composition of the vapor might be, and Vapor Management accomplishes this with little or no control of the cooling water supply. In both of these last two methods, **the volume of the product will dwindle towards the end of a run**.

With both Cooling and Vapor Management you will therefore have not just one indication of the onset of "tails", but two: rising temperature, and rapidly decreasing rate of production. If you miss these signals, the lower rate of production means that you will get less contamination than with Liquid Management techniques. That can be a huge advantage!

Conclusion

By examining: **how** fermentation works, then
how distillation works, and finally
how each component of a practical still works,
we've arrived at conceptual designs for a variety of systems, with confidence that they will operate efficiently and reliably.

Every part is there for a purpose, and we can say exactly why it's there. When you read Chapter 8, you will have an in-depth knowledge of how it all works and will be able to use that knowledge to develop your own designs and improvements.

We believe that **HOW** is the most important aspect of any hobby or pastime, and that there isn't anything that can't be improved through understanding and a little ingenuity. Above all, trying out your own ideas is **FUN**!

CHAPTER 5

OPERATING PROCEDURES

General principles

Before starting any job, it pays to think about what you want to do, and whether you can do it with the tools and materials you have. It would be nice to be able to duplicate any commercially produced beverage at will, but is it realistic? Unfortunately, the answer is no, because many factors are involved in creating the specific flavors in a product. However, you can duplicate many, and come close to many more. In this chapter we discuss how to operate the three types of still to produce different types of beverages.

A fermented product is a very complex mixture, containing dozens, if not hundreds of different compounds, and the distiller's task is to extract only a few. The fact that different compounds have different boiling points makes this possible, but it is very difficult to produce a specific mixture of a few components from such a complex starting point.

The boiling point of a mixture changes with the proportions of the mixture. This fact of nature determines the results obtained with each type of still. A simple pot still produces vapor that is rich in **all** the more volatile components, but does little to separate them from one another. The slight separation you do get can be increased by careful still design. An important element of a pot still is how vapor is transported from the boiler to the condenser. A traditional whiskey still has a gently curving dome with a long, narrowing, **Swan's neck** (also called a **Lyne arm)** to the condenser. Vapor condenses on the inner surfaces of the dome and the upright part of the swan's neck, creating some reflux and increasing the proportion of volatiles in the product. A simple pot still with a long, un-insulated upright tube to carry the vapor from the boiler to the condenser will give a slightly stronger product than one with a short tube. Adding a fractionating column will enhance the separation enormously by increasing the amount of exchange between vapor and condensed liquid. Adding a special condenser to increase and control the amount of reflux provides the best possible separation, allowing each component to be collected separately.

From our point of view, as small-scale distillers, the major difference between the different types of still is their relative ability to separate substances from one another. The fractionating column is much better than a pot still at doing this, and the compound column is as good as it gets.

The first substances to come out of any still are the most volatile compounds. Many of these are toxic substances, and they should be discarded or saved for another use, like fondue fuel. This first "family" of compounds is usually quite small, giving way quickly to a larger group of volatiles that arrive with the first part of the ethanol. This family, called the **heads**, contains some compounds that add flavor to spirits like whiskey, brandy or rum, but are undesirable in a pure ethanol product. The tails, which follow the main bulk of ethanol, contribute flavor, but also contain the toxins responsible for most hangovers.

If you want to try and make true flavored spirits like whiskey, brandy or rum, you need to keep both the heads and the tails and add a proportion of them back to the ethanol before aging. The small amounts of heads and tails will be altered by the slow chemical changes that occur during maturation in wooden casks. These compounds, along with flavors extracted from the wood, provide the flavor and body of the final product. Aging is the key process in the production of traditional spirits, and you may want to try it. Be warned, though – it takes years, skill and the correct environment to mature a fine spirit. Raw spirit tastes and smells nothing like the matured product, and the unaltered congeners in it are toxic headache producers.

Because making a good quality whiskey or brandy is so difficult, we will concentrate on the procedures required to isolate and collect the purest ethanol possible. If you want to flavor this, many rapid and simple options are available. There are many excellent essences on the market, and in the next chapter we'll show you how to make your own. After you have gained experience, you might want to try making a traditional product. Several good books on that advanced subject are listed in Appendix 8.

We will discuss the pot still, the fractionating still and the compound still in order, all using the same 30 liter (8 US gallon) boiler, containing a 20 liter (5.3 US gallon) batch. All the quantities and times mentioned in our examples are based on this 20 liter batch. 30 liters is a common size for a hobby still, but larger and smaller ones will work well. If you have a different size boiler, your quantities and times will change in proportion to the batch size. Boiler size has no effect on the dimensions of the rest of the still, which are determined by the laws of physics. A fractionating column needs to be 100 - 120 cm (3 – 4 feet) long, and its performance depends on heat input, not liquid capacity.

Doing two separate distillation runs can significantly improve the product. Commercial distilleries often do three! The first is a simple pot distillation to remove the solids color and much of the water that are in the fermented brew. The product collected in this run will be strong at first (around 60%), and will drop to zero as the run proceeds. The final average strength for the first run's product is usually 40%.

With very large batches (e.g. commercial whiskey stills) it's worthwhile removing some heads and tails at this first stage, but it's really not worth the trouble with batches smaller than 50 liters. If you have a boiler this big, you may find it useful to try.

If you are starting with a special ferment that containing 20% ethanol (see Chapter 1), then the first run can be omitted, but the benefits of higher concentrations are clear from the equilibrium charts in Chapter 2.

In the following examples, we will assume that several first runs have been saved up, allowing us to fill the boiler with 20 liters (5.3 US gallons) of impure 40% ethanol. This solution contains 8 liters of pure ethanol mixed with 12 liters of water and other compounds.

Pot Distillation

A pot still is the simplest type of still to make and run, but the purity and strength of the product are lower than achieved with other designs. Almost any size of tubing can be used to carry the vapor to the condenser, because the dimensions are not critical. 50 mm (2 inches) in diameter by 50 cm (20 inches) is a reasonable size. In this size still, the length or insulation of the tubing have a tiny effect on the quality of the product. You can easily experiment with tube length and insulation to see if you can tell any difference.

Stage 1

The 20 liters of 40% ethanol is brought to a boil as rapidly as the boiler can be heated. You can distill as rapidly as possible (like in the first runs) to save time, but it pays to reduce the heat to produce a gentle boil.

The ethanol concentration in the distillate will start out as high as 80%, and fall steadily through the run. The final products will be about 11liters (2.9 US gallons) of 60% ethanol, 2 liters (½ US gallon) of contaminated ethanol (the heads and tails), and 7 liters (1.8 US gallons) of dirty water left in the boiler. (If your boiler uses an immersion element, be sure its mounted low enough that it's still covered!)

The first 50 ml or so of product should be discarded or saved for fuel, because it contains toxic (and bad-smelling) compounds. This material is traditionally known as the **foreshots**, because it appears first, and at a significantly lower temperature than the ethanol.

The next 500ml or so will consist of the **heads**, which is mostly ethanol contaminated by enough foreshots to smell and taste. How much you discard is up to you, but the less you discard the less palatable the product will be. The best procedure is to save it all and add it to the next batch, because it is mostly ethanol that can be recovered by distillation.

Stage 2

The next 11 liters will be reasonably pure ethanol, mixed with water and some congeners. As the ethanol begins to decrease, the congeners get stronger, producing the tails. Again, your choice where to stop collecting the main product. You are facing a tradeoff between quality and quantity – you can have one or the other, but not both. Like the heads, the tails can be added to the next batch for re-distillation.

How long will all this take? For those who keep the heat full on and accept the consequent low quality, very little time indeed, as little as a couple of hours. Those who turn the heat down to 750 watt will take just over five hours to collect a higher quality product.

Stage 3

Some people like the taste and smell of the product as it is. It's similar to Irish Poteen or backwoods Moonshine, each of which has a dedicated following. Unless you are attempting a traditional product, you will want to get rid of the congeners that remain in the product. You can do this by distilling it again or treating it with activated carbon. (Instructions for using activated carbon are in Appendix 4.)

The final step is to reduce the product's strength to 40% or below with distilled water. You may try mixing the product with essences to make robustly flavored spirits, but we feel that the product of a pot still is generally not suitable for delicately flavored liqueurs.

Fractional Distillation

Replacing the connecting tube with a fractionating column significantly improves the separation of ethanol from water and congeners. The procedures are much the same as with the pot still, but a thermometer becomes a very useful tool, giving an indication of the boundaries between the heads, the body of the product, and the tails.

Stage 1

Again, the boiler is brought quickly up to temperature and the power reduced to about 750 – 1000 Watts. This lower power is very important, because the fractionating column exists to create the opportunity for exchange between vapor and refluxing condensate. The tradeoff between speed and quality is much more pronounced in a fractionating still.

The foreshots begin to appear at about 65-70ºC (149-158ºF) and are again discarded. The quantity of heads drops to 250-300 ml because the fractionating column produces purer fractions.

Stage 2

When the temperature of the vapor reaches 78-79ºC (172-174ºF), it is time to start collecting the main product. The temperature will stay stable at this level throughout most of the main run. The product can contain 90% ethanol if a very low heat setting is maintained and the column is well insulated.

Let's take a look at how the fractionating column affects the time taken to process the 20 liters of 40% ethanol. The pot still took just over 5 hours to produce 12 liters of 60% product (35ml/min at 750 watts. With the fractionating still, it takes around 2½ hours to collect just over 8 liters of 90% product. The same heat setting that produced 40 ml/min in the pot still produces 55ml per minute in the fractionating still. Half the time for a stronger and cleaner product! This is because the action in the column transfers heat from water to the more easily vaporized ethanol. Since the energy needed to vaporize water is 2.6 times greater than that for ethanol (540 cal/gm vs. 204), this heat transfer results in the production of more ethanol and less water. Given the two products (12 liters of 60% vs. 8 liters of 90%), the fractionating still is 2.3 times more energy efficient than the pot still! This theoretical calculation ignores the heat losses from the uninsulated pot still, which increases the relative efficiency of the fractionating still.

Stage 3

Toward the end of the run, the vapor temperature begins to rise slightly, and tails begin to appear in the product. Your nose is a better detector of tails than the thermometer, though, and toward the end of the run it is best to start collecting into a bottle and check for the presence of tails every ten minutes or so. If there are no tails, add the contents of the jar to the bulk container each time you check. Once tails appear, collect into a separate container until the temperature begins to rise rapidly, signaling the end of the run. If you desire some of their flavor, you can add small amounts of the heads and tails back to your batch, and the rest can be added to the boiler with the next batch, to recover the ethanol they contain.

Stage 4

You may well find the product satisfactory straight out of the still, but if it needs further purification, you again have the choice between activated carbon treatment or another round of distillation.

Compound Distillation

If you're on the quest for the purest product that can be made by distillation, the compound still is your tool. The price of the distinctly higher purity is a slightly more complex operation and longer operating runs.

The compound still is simply a fractionating still with a condenser on the top and a means of returning controlled amounts of condensed liquid back to the column as **induced reflux**. The fractionating still achieves reasonable separation through the naturally occurring reflux in its packing, and the longer its packed column the better it works. The compound column accomplishes significantly better separation to occur by recycling the reflux in the upper portion of its column over and over again, until the components of the liquid/vapor mix in the column settle into **dynamic equilibrium**.

Stage 1

It is extremely important to give the column enough time to equilibrate. When this state is achieved, the composition of this mixture remains the same in each zone of the column, even though molecules are entering and leaving that point constantly. In particular, the liquid/vapor mix at the very top of the column will eventually consist of one, and only one compound. However, this will **only** be true when **all** the vapor reaching the top is returned to the column as liquid reflux. As soon as some of the condensed vapor is collected as product, then other components will rise a bit higher in the column. This has been covered in previous chapters, but it is worth repeating here, because understanding this is vital to the correct operation of a compound still.

Once the boiler is at temperature, and the power controller is turned down to around 750 watts, make sure that **all** the condensed liquid is returned to the column, and that the condenser on top is condensing all the vapor. The column is then left alone for two hours, while it reaches equilibrium and **all** of the most volatile component has had a chance to make its way to the top.

Many people worry that if the reflux is cooled too much, it will upset the equilibrium in the column, and many ingenious designs have been proposed to minimize this "super-cooling". Fortunately, these fears are groundless. What happens when the reflux is cooled below its condensation temperature (which is very difficult to avoid if you want to condense all the vapor) is that a "buffer zone" is established in the very top layer of packing. Here, the cool reflux is quickly warmed up to its boiling point, and both equilibrium and the overall reflux ratio of the column are maintained. See Chapter 8 for a full discussion of how this works.

Stage 2

After two hours have elapsed at 100% reflux, the system is in equilibrium, and the top section filled with the foreshots. You need to collect this product very slowly, (1-2 drops per second), so you will not disturb the equilibrium. You must proceed cautiously at this stage, because there is only a small quantity of the foreshots and it would be easy to disturb the small zone they inhabit in the top of the column. You will begin collecting them at a temperature as low as 60ºC (140º F), and as they are collected, the temperature will slowly rise. Discard them until the vapor temperature stops rising, and then begin collecting the heads, which may be added to the next batch for ethanol recovery. By this time, the temperature should be stable at about 78º C (173º F). You can expect 100 - 150 ml of heads from an average batch. Your nose should be the best judge of when the heads have all been collected, but if you wish to be absolutely sure (or if you have a cold!), collect 250 - 300 ml (about a cup).

Stage 3

From this point on, an almost pure azeotropic mix of ethanol and water will be coming through and may be collected as the main product. The temperature should remain absolutely stable throughout the collection of the main product, and you can increase the rate of collection to around 4 drops per second.

We give guidelines in terms of drops per second, but the size of a drop is affected by many things – its composition, its temperature, and the size of the tube it is dripping from. The real measure is the production rate, remembering that we want to collect the main body of ethanol at 90% reflux. 750 Watts will produce about 50 ml of 95% ethanol per minute, so you want to be collecting between 4 and 5 ml per minute to establish a reflux ratio of 90%.

The best way to measure this is to establish a slow flow, begin collecting it in a small graduated cylinder, and measure the time it takes to collect 10 ml. Keep adjusting the valve until it takes two to two and a half minutes. When you are satisfied with the collection rate, count the number of drops you get in 10 seconds. Do this three or four times, and take the average.

In the future, you can reset the still to the same 90% reflux ratio by adjusting the valve until you have the correct number of drops falling in 10 seconds. This adjustment may be made quite rapidly.

In our experience, the correct rate of collection is found at about 35 drops in 10 seconds – not far off the 4 drops we recommended above! For the best accuracy, though, you should make this measurement yourself and calibrate your system.

Calculating on the basis that the 20 liters put in the boiler contains 8 liters of ethanol, it will take around **14** hours to collect all of it. You could get slightly better purity if you reduced the draw-off rate to 2 drops a second, but the run would take twice as long.

To achieve the highest purity, you will take the slow road, and you probably won't want to watch the still for 16-18 hours, not to mention the extra attention required near the end, checking for the onset of tails. Fortunately, all the toxic compounds and heads have been removed, so it's perfectly safe to stop the run at any time and resume it later. You will have to equilibrate the column again, but since the most volatile component is now ethanol, it will only take an hour at 100% reflux. Once equilibrium is achieved, you can start collecting product immediately.

Stage 4

Since you know how much ethanol was in the boiler, and you know the rate of collection, you can calculate when the end of the run should be. About an hour before this time, you should start collecting in small bottles and checking them for taste and smell before adding them to the main container. Your sense of smell is much more sensitive than the thermometer.

When you detect tails in the product, switch to a tails container. You can raise the collection rate now, and continue until the temperature has risen a few degrees, indicating that all the ethanol has been collected. You can add the collected tails to the boiler of the next batch along with the heads.

Stage 5

The product from a compound still is often much purer than commercially produced vodka, and most people are very satisfied with it as it comes from the still. If you want the ultimate in purity, process the product with activated carbon to remove the last hint of any congeners. We especially recommend carbon treatment if you are planning on making perfumes, essences, or delicately flavored liqueurs. It shouldn't be necessary if you want to make strongly flavored beverages or mixed drinks. The final choice, of course, is up to you, and you should have some fun experimenting with these options. Let your own taste and preference be your guide.

CHAPTER 6

BOTANICALS AND ESSENCES

The Flavors and Scents of Nature

So many excellent essential oils, essences and extracts are readily available that you might ask why we bother with a chapter on how to extract them yourself. Two good reasons come to mind. First, water distillation of essential oils is legal everywhere, so this is a type of distillation anyone can practice, wherever they live. Second, the production of essential oils, essences and extracts is an interesting and challenging hobby in its own right.

The prohibition against distilling ethanol is a very important legal consideration in some countries, and we can't stress too highly that the **ethanol extraction techniques described in other chapters may not be performed where it is illegal**. Other hydrocarbon solvents may be used, such as acetone and hexane but they are toxic, and since incomplete separation of the solvent would ruin the flavor of essential oils, we prefer the simplicity of steam distillation. Indeed, water is often better than ethanol for this purpose as its boiling point is higher. Where ethanol **is** used in this book, it's **solely** for its passive solvent properties to extract or carry oils and aromatic compounds in the form of an essence, tincture or extract. These can be legally made with purchased ethanol.

What are essential oils, and where are they found? Technically, an essential oil is a water-insoluble aromatic substance that can be extracted from botanical materials with heat or solvents. In practice, the term "essential oil" is sometimes loosely applied to all flavoring or aromatic compounds, when in fact many of these compounds are not oils at all. A familiar example of a non-oil aromatic is vanilla. The vanilla bean pod is green when harvested and must be cured by fermentation and drying, which can take up to 6 months. The principal flavoring compound is vanillin, which may crystallize on the dark brown pods as they dry. Over 150 other flavored compounds are found in the pods, and they contribute to the depth, flavor and aroma of the real vanilla. Vanillin is not an oil, but a substance called a "resinoid" or "oleoresin", and is extracted from the pods together with many other compounds by soaking them in a solvent (usually ethanol) to make an essence or extract. One of the first projects we suggest you try is making your own vanilla extract. Once you've experienced the true flavor and aroma of real vanilla, you'll never want to use anything else!

There are many terms used for plant extracts, and the techniques for making them. We have assembled a list terms you are likely to encounter as you become familiar with extracts, essences and oils. Since making extracts and oils is an ancient art, often different terms are used for the same process or product.

Definitions

Alcohol extract: Herbal materials are soaked in alcohol and then filtered out. The alcohol retains essential oils, resins and other compounds. Extracts are equivalent to **essences** and **tinctures**.

Absolute: A solvent extract of fragrant materials from botanicals, producing an alcohol-soluble liquid or semi-liquid oil. Common solvents include, among others, ethanol and hexane, which are then removed.

Attar: A traditional term for the steam-distilled essential oil of rose petals. This term is also used in India for the material obtained by distilling rose petals and sandalwood together.

Balsam: A resinous, semi-solid mass or viscous liquid exuded from a plant. A "true" balsam is characterized by its high content of benzoic or cinnamic acid or their salts. They are often associated with pathological or physiological products.

Cold pressed: a pressing process of extraction for citrus and fixed oils with minimized heat and deterioration, usually under 49ºC (120º F). The same thing as **expression**.

Concrete: a concentrate, waxy, solid or semi-solid perfume material prepared from previously alive plant matter, usually using a hydrocarbon solvent, which is then removed.

Distillate water: Otherwise known as floral water or hydrosol, produced during steam distillation.

Enfleurage: A process for making perfumes in which odorless fats or oils absorb the fragrance of fresh flowers (from French: *enfleurer*, to saturate with the perfume of flowers).

Essence: An alcoholic extract (see Tincture). This term is also used commercially for prepared mixtures of flavorings used to prepare beverages and liquors.

Essential oil: Volatile oils, typically fragrant, which are extracted from botanicals using steam distillation or expression. Essential oils are normally liquid, but some cases, like anise, may be solid at low temperatures.

Expression: The extraction of essential oils and non-volatile materials by pressing the natural material.

Extraction: a process of removing botanical components from a raw material through the uses of distillation, solvents, heat, or pressure. The extract will contain non-volatile as well as volatile components. Oleoresins, resinoids, concretes and absolutes are all produced by extraction.

Exudate: A non-cellular, natural raw material that is secreted by plants, either spontaneously or after wounding. Balsam Peru and Balsam copaiba are examples of exudates.

Fixative: A material that slows down the rate of evaporation of the more volatile components in perfume and natural products formulation.

Fixed oil: A name given to a vegetable oil obtained from plants that, unlike essential oils, are fatty, dense and non-volatile. Olive, peanut and corn oils are all fixed oils.

Gum: A water-soluble exudate consisting mainly of polysaccharides, used principally as a thickener.

Gum resin absolute: An oil-soluble, purified exudate consisting mostly of resinous constituents, gums and small amounts of volatile components. Examples are Myrrh, galbanum and opoponax.

Hydrosol: Water containing dissolved volatile substances from plant material. The same as distillate water.

Infusion: Extraction of materials from plant matter by soaking in cold or warm liquid solvents, or in oils or fats. Tea is a water infusion of camellia leaves.

Maceration: Soaking until soft.

Oleo gum resin: A natural exudates from trees and plants that consists mainly of essential oil, gum and resin.

Oleoresin: A natural resinous exudation from plants, or an aromatic liquid preparation, extracted from botanical matter using solvents. They consist almost entirely of essential oils and resins.

Pomade: A prepared perfume material obtained by the enfleurage process.

Rectification: A second distillation of an essential oil to remove color, resinous matter and perhaps unwanted flavors.

Resin: A natural or prepared product, either solid or semi-solid in nature. Natural resins are exudations from trees, such as mastic; prepared resins are oleoresins from which essential oil has been removed.

Resinoid: A perfumery material prepared from natural resinous matter, such as balsams and gum resins, by extraction with an hydrocarbon solvent.

Tincture: An alcohol extract.

Essential oils

Whether or not you know it, you encounter essential oils every day of your life, since they are widely used in food, candy, beverages, perfume, soap and cosmetics.

Essential oils were well known even in ancient times. The earliest references to them come from India, China, Persia and Egypt, and the Greeks and Romans conducted extensive trade in oils and ointments throughout the known world. They were extremely valuable products, often worth their weight in gold. This is explains why frankincense and myrrh were considered such valuable gifts in biblical times, though they're quite inexpensive today.

Most of the ancient products were probably prepared by long soaking of flowers, leaves, or roots in vegetable oils, or by enfleurage, because distillation was unknown. Arabian alchemists in the middle ages invented distillation and discovered the solvents that allow production of the essential oils we know today.

Knowledge of distillation spread to Europe, and essential oils became a specialty of the medieval pharmacies. By the middle of the 18th century about 100 essential oils had been discovered, and in the early 1900s, better technologies greatly expanded their production.

Natural essential oils are not cheap, and some of the rare or difficult to extract ones can cost several thousand dollars a pound. The high price and limited availability of natural oils encouraged chemists to search for substitutes. The major constituents of many have been synthesized and are now in everyday use. The commonest example of a synthetic flavoring is vanillin, which appears in literally millions of prepared foods. Most of the "fruit flavors" listed on labels are actually synthetic esters. The ready availability of cheap synthetic flavorings has expanded the number of food choices, but they lack the subtlety and of natural flavorings, which comes from the large number of compounds found in addition to the "primary ingredient".

Today, essential oils are usually extracted by steam distillation. There are three basic methods of steam distillation. The oldest and simplest method is to place chopped or crushed plant material in water, bring the water to a boil, and recover the oil and a hydrosol by condensing the vapors. This method is still used on a very small scale in laboratories and homes, but is very inefficient on an industrial scale. The more modern technique boils water in a separate vessel and passes steam through the plant material, which is held in a special chamber. The oil and hydrosol are collected in the same way. The third variant separately heats the plant material in its chamber so no steam will condense on it, achieving "dry" distillation. This can be useful if you are extracting plant material that will swell up or clump together when it gets wet.

Steam distillation is not the only method used to isolate essential oils. Where the oils are plentiful and the source is relatively soft and easily accessible (like citrus peels), the oils are often literally squeezed out, in a process called **expression**. The material is squeezed in huge presses and the oil either drips out or is extracted from the crushed mass with solvents.

Certain delicate oils are obtained by solvent extraction with as little heat as possible. When particularly strong extracts are required, removal of the solvents can yield a pure oil or a solid waxy substance called a concrete. Adding a little solvent to a concrete can make a semi-liquid material called an absolute.

Methods of Extraction

We mentioned vanilla as an example of extraction by soaking in a cold solvent, but this is not always the best way of extracting the flavored and scented substances found in the fruits, seeds, leaves, bark or roots of plants. The list of methods below is not exhaustive, but does cover the methods that you can safely use at home.

- Cold soaking at room temperature in a solvent such as water or ethanol.
- Hot soaking by subjecting the plant material to a hot liquid solvent.
- Boiling the plant material in a solvent, or passing hot vapor through the material to vaporize the substances required. **Note**: we only recommend steam for this method.
- Expression, or squeezing to extract under pressure.
- Infusion into a solid or semi-solid "carrier" material such as fats or oils, followed by extraction.

Cold Infusion

Cold infusion (soaking) is the simplest technique, and has the advantage of not using heat. Many large organic molecules break down when heated, with considerable changes in their flavor and aroma. Jasmine is an example of a plant material that cannot be successfully extracted with hot solvents or steam. Cold soaking is a great way to start extracting botanicals, because the only equipment you need is a large jar or bottle.

Fig. 6-1

Cold soaking is used widely in Europe to prepare some excellent liqueurs. In Germany, the "Rumtopf" or "Rum Pot" is a Christmas tradition. Early in the year, about a kilogram (2.2 pounds) of the first berry fruits are put in the bottom of a big ceramic pot together with a kilogram of sugar. A bottle of rum is poured on top and a close-fitting lid put on the pot to prevent evaporation. As different fruits come into season throughout the year, they are added, along with more sugar and rum as needed. When Christmas arrives, the pot is opened to yield a rich, ruby red fruit liqueur (without a hint of rum flavor), and a superb supply of fruit for desserts.

Other familiar products produced this way are the herbal and flower-flavored oils and vinegars that are now widely available and easily made.

Here is a recipe for the vanilla essence that we mentioned earlier: Cut several vanilla pods into small pieces about I cm ($^1/_2$ inch) long, place them in a small screw-top jar, and fill it with 95% ethanol. Allow it to soak for at least two weeks. The longer this is kept, the better it gets.

If you can't make your own 95%, it is sold in the U.S. as "Everclear", and in Europe as "Polish Spirit". Just make sure that any strong ethanol you buy is intended for beverage use and does not contain de-naturants!

Hot Infusion

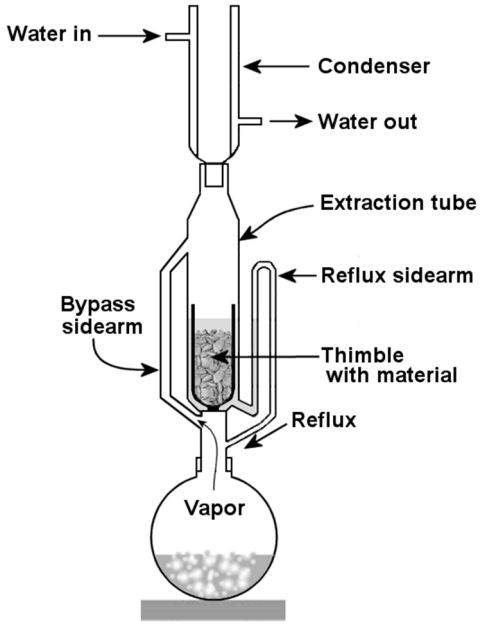
Hot soaking in a water or a hydrocarbon solvent becomes necessary when some of the compounds you want to extract are not volatile, slow to dissolve at room temperature, or are bound up in dense material. Solvents become more powerful as the temperature rises. Hot infusion of leaves, bark, seeds or roots is exactly the same as process as making coffee or tea. You can, in fact make coffee or tea with cold water, but the extraction takes several hours, rather than a few minutes with hot water. In the same way, many botanical materials will extract rapidly with added heat, but might take weeks at room temperature.

When making a hot infusion, you must be careful that the more volatile compounds don't evaporate. This is **not** distillation, and the temperature should not reach the boiling point of either the solvent or the volatile compounds being extracted.

A form of extended hot soaking is used in the preparation of Italian walnut liqueur. Green walnuts (before the shells harden) are packed into large jars and covered with sugar, herbs and grappa at about 80% ethanol. The sealed jars are then placed out in the full sun, where they get quite hot every day. In a few months the contents mature to a delicious, dark liqueur. The water in unripe walnuts reduces the strength of the spirits from around 80% to a final strength of about 40%.

Hot soaking is a technique that's worth trying when cold soaking is ineffective. In general, you should try the simplest method first – i.e. try cold soaking before applying heat. Experiment with small quantities before committing yourself to a large batch!

As compounds begin to dissolve in the solvent, they reduce its capacity to dissolve any more of the compound. Many sparingly soluble compounds require so much solvent to extract them that doing so is impractical or uneconomical. Franz von Soxhlet was a German chemist who solved this dilemma, and the device named after him (the Soxhlet extractor) is a mainstay of analytical laboratories to this day. Like most clever inventions, it is startling in its simplicity, and once you understand how it works, it is easy to make one of your own.



The solvent is boiled in the small container below, and the vapor travels upward through the bypass tube into the extraction tube and then into the condenser. The hot condensed vapor then drips into the extractor body, which contains the sample. If the material being extracted is crumbled or powdered, it is usually held in a "thimble" made of some porous material, so the solvent can pass through it.

As pure, hot solvent drips into the extractor body, the level of liquid rises until all the material is submerged. The compounds we are extracting begin to dissolve in the pure solvent, and the liquid level continues to rise until it reaches the top of the "U" tube outside the main body. When this happens, a siphon is established, and all the liquid drains back into the boiling flask.

Fig. 6-2

The genius of the soxhlet extractor is that it constantly re-purifies the same small amount of solvent (by distillation!), and re-uses it. The extracted materials are kept in the boiler while the sample in the extraction chamber is constantly bathed in fresh, pure, hot solvent. This greatly increases the extraction rate and the concentration of the final product.

Needless to say, volatile compounds do not need the kind of concentrated attack provided by the soxhlet extractor, but it does an excellent job of rapidly extracting difficult compounds, and is particularly useful when dealing with small quantities, such as seeds.

Boiling

When neither cold nor warm soaking is fully effective, heating the solvent to its boiling point often does the trick. This is particularly so when the flavor and aromatic compounds are tightly bound in a dense matrix of plant material which hinders access by the solvent. Seeds, bark and root material can all fall into this category and extraction using a boiling solvent is often the only practical way of getting a good yield. This process is limited to use with products that are not affected by heat, though. Once again, it makes good sense to experiment with a small quantity before attempting a large-scale extraction. Many chemical and herbal handbooks contain lists of compounds and botanicals that are either resistant to, or destroyed by heat.

A technique that can work well with heat-resistant compounds is to boil plant material in a solvent and collect the distilled vapor. The argument is that the act of distillation will "purify" the product, only the most volatile compounds being carried over in the vapor. This is one of the traditional methods of making gin and many liqueurs. Curacao, for example, is the distilled extract of bitter orange peels.

Since many plant materials are degraded by heat, the results are not always what you might hope for. The best advice we can give is to compare the results you get with methods of gradually increasing severity, and choose the one you like best

Extracting with liquids creates a dilemma: some compounds require high heat or extended time to fully extract, while those very conditions can ruin some desired compounds or extract unwanted ones. There are some materials that you simply can't successfully extract with liquids.

Steam distillation

Volatile materials, like essential oils, evaporate in the presence of heat. Even compounds that are sensitive to high temperatures can withstand them for short periods of time. Steam distillation is a process that produces localized high temperatures for a short period of time, and very effectively extracts materials like essential oils.

The simplest way to practice steam distillation is to simply place the materials in a small pot (like a coffee carafe) with water, collect and condense the steam and vaporized oils and then skim the oils off the surface of the water. With some materials this will work quite well, but many delicate botanicals will not stand up to this treatment.

A more sophisticated method is "dry" steam distillation. In this process, steam is passed through a loosely packed mass of the plant material and a considerable amount of heat is liberated when the steam condenses to water. Every gram of water that condenses on the plant material releases 540 calories of heat at a temperature of 100º C (212º F), which is more than enough to vaporize most plant oils. Often those oils are bound inside the plant material in sealed pockets. When heated rapidly, the oils expand, break the walls of their prisons, and escape.

The released oils, in the form of aerosols and vapor (the oils boil at a lower temperature in the presence of steam than on their own – see Chapter 8, Raoult's Law), are swept up by the passing steam and carried to the condenser, where they're abruptly cooled down. The steam is condensed to water, and the extracted oils float on the surface of that water. Some of the extracted compounds are soluble in water, and these are collected as a **hydrosol**.

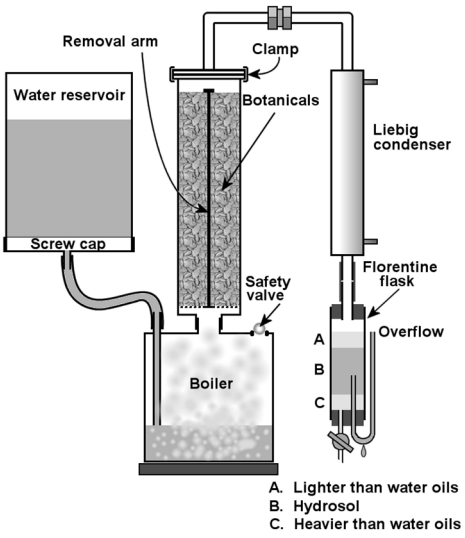


Fig. 6-3

We have designed a home-scale steam distillation apparatus that can be used for simple or "dry" steam distillation.
This diagram is not to scale, but the device is so small and simple that detailed plans should not be necessary. Let's examine how it works, section by section.

Water can be fed to the boiler from a reservoir. There are three reasons for this:

- To keep a small amount of water in the boiler, so it can come to a boil quickly.
- To assure that the boiler always has the same amount of water in it.
- To give you a visual check that there's enough water and that you won't run dry.

You will also note that there is a "safety valve". Hopefully, this will never be needed, but it is a prudent feature to have, particularly because it's so simple: a marble sitting on a rubber grommet placed in a hole through the boiler lid!

However, this reservoir feature is **not** a necessity. A pressure cooker with a hose fitted to the steam nozzle can make a fine boiler (just like the basic pot still), but it takes longer to heat up and you must to put enough water in it for the whole run. Fortunately, most small-scale steam distillation runs are quite short. Commercially available pressure cookers are equipped with safety valves, but these operate at quite high pressures. If using a pressure cooker that you don't want to drill holes in, it would be prudent to add to the delivery tube a little side-arm carrying a marble safety valve.

Steam passes to the plant material tube or chamber, which is the key to efficient extraction. This may be any size you like, but we find that between one and two liters is more than sufficient for most needs. Descriptions of industrial-scale steam distillation often speak of materials being “suspended in baskets” in the still. They do this so that they can change the depleted materials for new ones quickly, but some of the steam may flow around the basket. On the home scale, a chamber is easy to fill with plant material and ensures that all of the steam goes through it.

Pack the plant material loosely in the chamber, chopping it into small pieces if necessary. There is a perforated plate or screen at the bottom to retain the material, which has a thin rod or wire attached to it, making it easy to extract the spent material after a run. If you are processing leaves or materials that clump together when subjected to steam, you need to mix in some other material to keep the whole mass porous. Small, uneven pebbles, Raschig rings, wood chips, etc., are all good possibilities. Make sure that they have been boiled or steamed extensively, so that they will not contribute any odors or flavors of their own to your product!

A copper tube passing through the tightly-fastening lid of the chamber leads to a simple Liebig condenser. Do not use polypropylene tubing for this as essential oils can soak into plastics. The condensed steam and the volatile oils then drip into a **Florentine collector**.

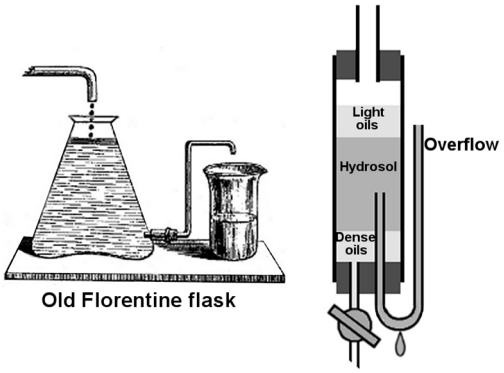


Fig. 6-4

Steam distillation produces a mixture of water and oils, and it is usually the oils that you’re trying to collect. The Florentine collector is an ingenious solution to this problem, and has been in use by the perfume industry for centuries. Let’s discuss how it operates with a couple of examples. The device is first filled with water (preferably distilled) until the bottom of the overflow pipe is covered (so no precious oil will block it). As the collector fills with condensed liquids, the oils and water separate out naturally, with lighter than water oils floating to the top and heavier than water oils sinking to the bottom. (Most oils are lighter than water, but a few, including anise, cinnamon, clove, nutmeg, and mace, are heavier than water and will sink.)

The overflow pipe is set up so only water enters it, and when the liquid level in the collector reaches the top of the pipe, it begins to overflow, keeping the level inside the collector constant. Since the density of the top oils is lower than that of water, the actual level in the collector tube may be slightly higher than the top of the outside arm. The water, or more correctly the **hydrosol**, is continually removed to a separate collection vessel. Depending upon the type of botanical, the hydrosol might contain substances you want. Hydrosols are widely used in making perfumes, soaps and cosmetics.

At the end of a run, collect the heavier than water oils from the collector through the stopcock at the bottom, then the small amount of hydrosol still trapped in the collector, and finally the lighter than water oils that float on top. You'll find that most of the oils will come across in the first half hour of steaming, but continuing to get the last bit for a further half to one and a half hours won't hurt. Oils will not get into the overflow tube, because it's always in water or hydrosol. Even when the product is withdrawn, hydrosol in the overflow tube will discharge back into the main collector, keeping the opening clear.

Steam-distilled essential oils are extremely concentrated and you should be careful not to let them touch your skin, and to handle them only in a well ventilated area. The yield from one batch (our botanicals chamber contains 2 liters of plant material) is only a few milliliters of oil, but these oils are extremely potent in their pure, undiluted form!

The simplicity and small size of the equipment make steam distillation an ideal first step into distillation in general, and it is a very interesting and rewarding pastime, too.

Expression

With some kinds of plant material, there's no need for solvents or heat at all. You can simply squeeze the flavoring compounds out of the plant material. Citrus peels are the best-known example.

Many of the intense flavors and scents we want are contained in the oils that permeate the outer surface (the **zest**) of citrus fruit. The white pith is bitter, so be careful to separate the thin, outer oil-bearing layer from it. You can do this with an ordinary kitchen grater, but a Stanley Surform ® Corner Plane is an even better tool. This small woodworker's tool not only cuts the zest off cleanly, it also collects the shavings neatly in a recess above the cutting blade.

Fig. 6-5

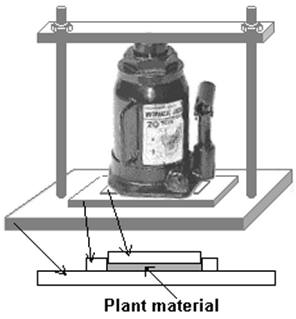


Fig. 6-6

The oils in the citrus shavings can be extracted either by soaking in a strong ethanol solution (see "Cold Soaking" above), or by subjecting them to extreme pressure. In this case, “extreme” means up to a tonne per square centimeter, (4 tons per square inch)! Surprisingly, you can generate these kinds of pressure easily at home, with a sturdy metal frame and a small hydraulic jack, as illustrated in Fig. 6-6.

The citrus zest placed on a strong metal tray, then a flat metal plate is placed on top of this and the jack placed on top. The jack and the plates are placed in the metal frame, the top bar adjusted to fit snugly, and pressure exerted with a few strokes of the jack arm. After 10 minutes or so, practically all the oils will have been squeezed out of the rind. Don't expect them to come rushing out in a flood, like oil from olives or juice from grapes, because the quantities are very small. Finally, wash the material into a container with a little vodka or 95% ethanol, straining out the zest, to leave a concentrated solution of the essential oils.

It may seem like a lot of trouble to obtain just a little citrus oil, but the flavors are so intense that you don’t need much.

Solid infusion (Enfleurage)

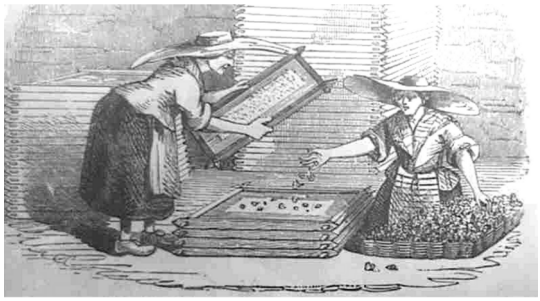


Fig. 6-7

Solid infusion is another very old method once used extensively by the perfume and cosmetic industry. It's the only really simple way can extract the scent from small quantities of flowers. The technique, known as **enfleurage**, is a process in which odorless fats or oils absorb the fragrance of fresh flowers. In the old times, both pork and beef fat were used, and the fresh-picked flowers were pressed against thin layers of the fat for a few days before being replaced by new, fresh flowers. The scent-laden fats were then dissolved in ethanol and the delicate perfumes were distilled off. It's an old technique and no longer widely used, but you might like to explore it if you're interested in making perfumes.

Be careful! This technique involves distillation of ethanol in its final stage, and may be illegal in your area! You may wish to try other absorbent materials such as Fullers Earth (a fine powdered clay-like material that absorbs oils) and then extracting the scent materials by **soaking** in 95% ethanol.

We have to frankly admit that we haven't actually tried this technique ourselves, but it is so unusual that we had to mention it. If you do try it, remember that hydrocarbon solvents (unlike pure ethanol) generally have an odor that will swamp the delicate flower scents.

The Product

Some of the products you get, particularly the hydrosols, will be cloudy or "milky" liquids. This is due to the fact that some of the products are very fine suspensions of concentrated substances. Some of them will clear on their own, but some cannot clear, by their very nature. Don't worry about it too much, because you will be using tiny quantities, and if you are using them to flavor spirits or scent perfumes, the substances will dissolve immediately when added to ethanol.

It's very rare that just one, single ingredient of a recipe provides the perfect balance of flavors, and this is as true of beverages and perfumes as it is in cooking. Prepared essences that claim to reproduce the flavor of gin, rum, whisky, cherry brandy, etc. contain a wide variety of ingredients, but still rarely provide the exact flavor you expect.

Many kinds of pure essential oils are readily available at reasonable prices. They are convenient, and are a good source of flavors and aromas to "mix and match" as you formulate recipes for beverages. However, simply because they **are** so potent and pure, you must be very careful in using them. Most successful perfumes, and many well-known liqueurs have dozens of separate ingredients to get exactly the right balance, and then often require a long time to "mature". Just like different brands of vanilla extract have subtly different flavors, so will essential oils from different sources. Triple Sec made from your own freshly pressed citrus oils will almost certainly taste different from one made with purchased extracts. Which one is better is for you to decide, and the experimenting to find out is at least half of the fun of this activity.

Don't be disappointed if your initial attempts to imitate any of the famous spirits or liqueurs fail. Start with small batches, and don't be afraid to doctor them and see how the flavors and aromas change. Improvement comes with experience, and a lot of experience can be had from one 250 ml. (1 cup) batch, if you experiment with it. Have fun discovering what you can do to the flavors, and who knows? You might just create a wonderful new product of your own!

CHAPTER 7

THE WORKSHOP

Fig. 7-1

You don't have to grow your own grapes make wine, nor do you need to make all of your own equipment to become a practical distiller. However, it is very useful to know how to make things yourself, even if it's just how to connect pre-fabricated parts together in different ways. In this chapter, we'll cover workshop basics and several small projects that will help you with your equipment.

We will cover the basic techniques needed to make equipment out of copper or brass, and techniques that can be used with stainless steel (silver soldering and brazing). We will not cover advanced techniques like glassblowing or welding, because they are difficult arts and require a lot of practice. If you want equipment made from stainless or glass, then make full use of ready-made parts and fasteners. For those bits and pieces you just can't buy, find someone who has the skills and facilities to do the job for you. Many home brewers have received considerable help for a couple of six packs!

Safety

Fig. 7-2

Working with metal creates many hazards to beware of. Cutting and drilling create sharp edges and burrs that can cause nasty cuts, and can cause small, sharp pieces of metal to fly around at high speed. Wrap-around safety goggles (which can easily fit over spectacles if you wear them) are a **must**! So-called "safety glasses" that permit objects to get to your eyes from the side can be worse than useless because they create a false sense of security.

Soldering metal pieces involves the use of very high temperatures that can cause second or even third degree burns in seconds. Heavy leather gloves are therefore a very worthwhile investment. You should develop the habit of feeling naked and unprotected whenever you don't have on the proper safety gear.

Beyond this, we don't want to belabor the point. Good safety equipment is no substitute for common sense! If you develop your workshop beyond the basics, then you will be bombarded with sound safety advice from other sources, be it from the manufactures of the equipment you buy, or from instructors at classes you may attend.

However, we'd like to add just one word of caution. In any activity or line of work, the most dangerous phase is after you develop skills and familiarity, and caution begins to lapse. Many people have been badly hurt or killed by bad habits that built up bit by bit as they gained confidence and experience. Be forewarned, and guard against this tendency.

Some basic requirements

What do you need to get started? A place to work, something to hold the pieces being worked on, and a few basic tools will allow you to make a surprising number of quite complicated things. Of course, the better the workspace and the tools, the easier many tasks will become. As with all hobbies, it is best to start with the basics and try your hand. If you are having fun and making progress, you will find it far easier to justify each acquisition of more and better tools and equipment.

The following list of tools is basic, and by no means comprehensive. You may need only a few of them, so we've tried to give the list some sort of "priority" based on what we find useful. Your priorities may be different, depending on what you're trying to do.

- Safety goggles
- Ruler, marked in both inches and centimeters.
- Hacksaw, with fine metal-cutting blade.
- Screwdrivers, both flat and Phillips head (cross-head).
- Hammer.
- Pliers – especially a set of waterman's pliers (slip-joint type)
- Leather workshop gloves (use them when handling hot components)
- Vice and/or a couple of vise-grip pliers.
- Hand drill, preferably a rechargeable hand-held electric drill.
- Files, as many different types as you can get.
- Adjustable wrench.
- Emery paper – rough and fine grades.
- Center punch.
- A set of good quality drill bits, both metric and inch sizes
- Hand-held propane torch
- Lead-free solder and flux.
- Wire brush.
- Carpenter's square.
- Wood saw.
- Vernier calipers (extremely useful for measuring tubing – cheap yet accurate digital ones are widely available).

Add to this one of those folding "Workhorse" benches if your space is limited. These are the type which feature a "vise top", and are sometimes called "mitre benches", with guides for angle cuts.

The hand drill is the only power tool you may need at first. If you take on many projects, you will quickly want to move up to a good quality drill press. By the time you get around to lathes, milling machines and welding gear, you'll probably be able to teach us a few things about workshop techniques!

One readily available tool that will allow an amateur to "fudge" a little or a lot is a Dremel® Moto-Tool. This is a very small handheld cylinder with a rotating shaft that spins about 30,000 rpm. Many cutting wheels, brushes, grinders of various shapes, buffers, etc are available. With a little ingenuity you can do almost anything with it. It's so compact, you can put it away in a drawer for storage.

Please don't rush out with this list in your hand and buy up the shop. Get tools as you need them. Before getting any new tool, ask yourself if you'll use it more than just once or twice, and if not, whether you can manage to do the job some other way with what you already have. When you buy tools, always get the best quality that you can afford. Cheap tools may seem attractive, but if they don't last, or can't do accurate work, they're a waste of time and money. Think laterally too: one of the most useful tools in the workshop can be a diamond-impregnated nail file. These are tremendous for fine work!

Basic techniques

Here are some useful basic techniques and some tips and tricks that can save quite a bit of time. Remember, it's not our intention to put you through a long apprenticeship before you can begin enjoying yourself. You don't have to be a cabinet maker to enjoy whittling a stick!

A distillation rig is basically a set of containers and pipes joined together, so most of the techniques that follow will deal with how to assemble these components. This is only a start, because every new project will present its own, unique problems. We've tried to follow the usual order of events – measure, cut, join.

Measurement

The Golden Rule of the workshop is: Measure twice, cut once. It sounds simple, but is profound. Even NASA has suffered from not observing this rule closely enough. Multi-million dollar projects have been lost because plans were misread and a small component was machined to the wrong size.

Always allow for error. This is never truer than when drilling holes. Drills tend to wander when they first meet the metal they're supposed to cut through, and once a hole is started in the wrong place, it's extremely difficult to correct. This is why a center punch was higher in the list than the drill bits. With a center punch, you can take your time and make sure that the drill will start cutting in precisely the right place.

Rulers and careful measurement are fine, but nothing beats holding two pieces together to check they really **will** fit, and to keep doing this as you **gradually** cut or file them to their final form. This is a "handcraft" hobby, not a production line, so if you are drilling matching holes, use the holes in one piece to guide you when drilling the second piece (see "templates" later on).

Connecting pipes and tubes

By far the simplest way of connecting pipes or tubing end to end is to use an off-the-shelf threaded coupling, or a plumbing union. Both of these consist of two separate pieces that can be soldered onto the ends of the pipes to be joined, and then they can be tightened together or taken apart easily with a simple wrench. Connectors have been made with almost every conceivable situation in mind, and you can save yourself a lot of work by looking to see if an off-the-shelf item will do what you want. You can also combine off-the-shelf items in clever ways to do many unusual things.

Flanges

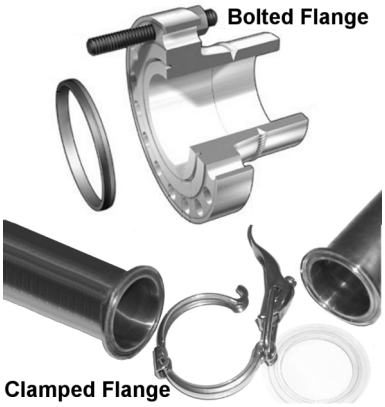


Fig. 7-3

There are times you need the extra strength of a flanged connection that is bolted or clamped together. The **flanges** that make this possible are relatively easy to make yourself.

A flange is just a flat rim on the end of the pipe or tubing, produced by soldering or welding on a flat plate with a hole in the middle that matches the size of the pipe. Flanges may be any shape, but round and square are the most common. Spring clamping flanges together may be enough to hold two pipes securely, but bolting them together provides a much stronger joint. Two types are most common, the 3-hole flange and the 4-hole flange.

3-Hole Flange

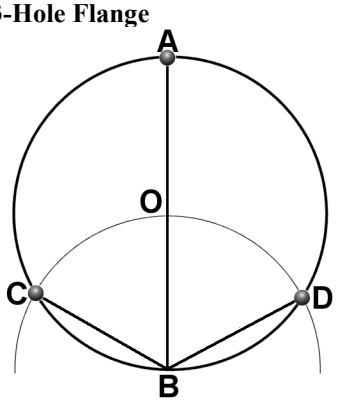


Fig. 7-4

If you draw a circle with a compass, then you can easily mark off three equidistant points for bolt holes. Just mark where you want one of the holes to be on the circle, then draw a line through there and the center of the circle, to meet the circle on the other side. Using that point as the center, mark off an arc of the same radius as the circle. It will meet the first circle at two points, and these will be where you drill the other two bolt holes. You can cut whatever size hole you like in the middle, using the center of the first circle.

4-Hole Flange

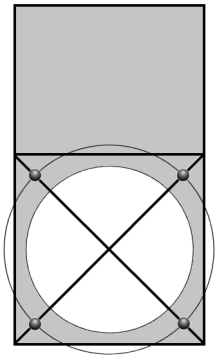


Fig. 7-5

4-hole flanges are even easier to make as you can get metal of almost any thickness you need in straight-sided flat plates. Mark off the amount you want to cut off so you end up with a square of metal, and find the center by drawing diagonal lines. Using your compass again, draw a couple of circles from that center, one to mark out the big central hole and the other to show where the bolt holes will lie. The bolt holes will be where the diagonal lines and the outer circle intersect.

Templates

We said before that you can save a lot of marking out by drilling holes in one piece, and then using those holes to guide the drill when cutting holes in the other piece. If you have to drill holes like this in a number of pieces, and you want them to all be the same, you can save a lot of time and trouble by carefully drilling small holes in a "master", or "template", then using that to guide you when drilling all the rest. You will find that you achieve surprisingly good accuracy if the guiding holes in the template are much smaller than the size of the final hole, and if you use the same small drill to make similar guide holes in each of the other "work" pieces. These small holes will help center a larger drill bit when you drill the holes out to their final size.

Cutting large holes

Cutting a large hole, (like the center of a flange) can be hard work. One simple way is often to mark off the large hole, then drill lots of small holes all the way round, just inside that circle. The center piece can then be knocked out and the hole finished by filing or by using a small grinding wheel in an electric hand-drill.

Other ways of connecting pipes and tubes

Ready-made couplings

Fig. 7-6

Hardware and plumbing stores carry a wide variety of off-the-shelf connectors that can save you the trouble of making flanges. Connectors have been designed with almost every conceivable situation in mind, and most are designed for easy assembly. Many connectors for pipes come ready soldered, and all that is needed is to slip them onto a pipe and apply heat to make a perfect joint. Others will be threaded, so you can screw the fitting onto a matching mate. These ready-made connectors are almost essential if you want to join stainless steel components, and many stainless couplings come with spring clamps to hold them together.

Compression couplings

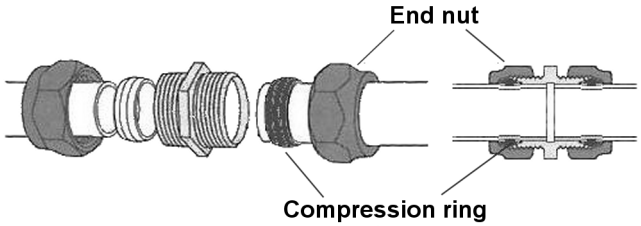


Fig. 7-7

One specialized type of connector is called a "compression coupling", and this is particularly useful for small diameter tubing. A small ring of metal with bevelled edges slips over the pipe. This is the "compression ring" (sometimes called an 'olive'), whose edges are forced inward to grip the tube when the hollow nut and bolt are screwed together. No soldering is needed, and it forms a gas-tight seal. The only drawback is that this is a one-time joint only, and you'll need another compression ring if you want to reposition the joint using the same nut and bolt.

Thermometer clamps

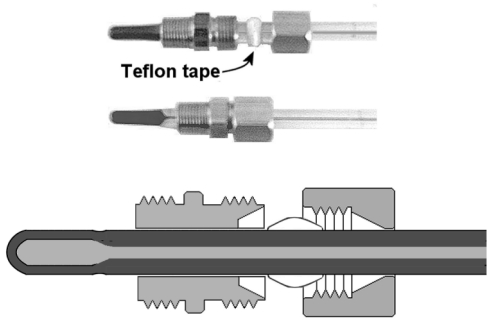


Fig. 7-8

With slight modification, these small compression ring connectors can be used to firmly hold even delicate items such as glass thermometers. All that is required is a compression coupling with holes in the nut and bolt that are the same diameter as the thermometer, or slightly smaller. If smaller, just drill the holes out so they fit on the thermometer snugly. Instead of the metal compression ring, make your own "gentle" compression ring by winding some Teflon® tape around the thermometer, between the hollow nut and bolt. This is squeezed together when the nut and bolt are tightened (only finger tight - you're dealing with glass!) and forms a gas-tight joint. The hollow bolt can be soldered into the side of a column before you introduce the thermometer. This is an ideal way to gently hold a delicate thermometer in place.

Joining small tubes to large pipes

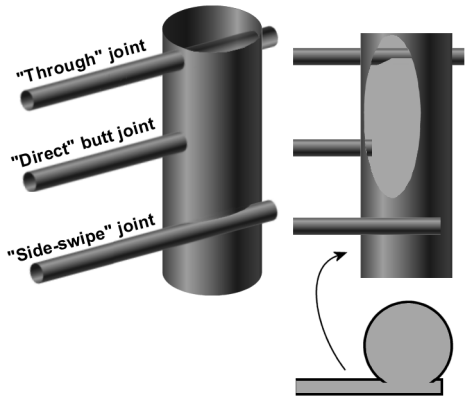


Fig. 7-9

You can't always find the perfect connector for the job, even if you use reducing couplers in combinations not anticipated by the manufacturer. This is often the case when joining small tubes to larger pipes and, in such cases, you'll have to make your own.

There are three simple ways to join small and large tubes together:

- A "direct" butt joint.
- A "through" joint.
- A "side-swipe" joint.

The "direct" butt joint is mechanically weak because it has very little area for support. The small tube should always pass right through an exactly matching hole in the wall of the large tube or plate to maximize the strength of the joint, and should be provided with a supplementary collar, or **fillet**, if possible. You can make a fillet by winding a couple of turns of copper wire around the small tube where it joins the larger one. When you solder them, the wire will hold extra solder and form a visible ramp of support. A compression ring secured to the small tube by tightening the matching nut and bolt, then removing them, makes an extremely neat "supplementary collar".

The "through" joint has the small tube passing completely through the large pipe and is soldered to both walls of the pipe. The small tube has half its side filed away before assembly to create the actual joint. This is a very strong joint, requiring no supplementary collar, and is preferred where the small tubing doesn't interfere with anything in the large pipe.

A "through" joint made with intact tubing makes an excellent "**thermowell**" for a thermometer if you don't want to use a compression coupling.

The "side-swipe" joint is the most difficult to make, because it requires careful filing of a groove in the larger tube, using a round file with the same diameter as the small tube. One side of the small pipe is filed away to match the opening in the large tube, and the stub end is blocked, usually by soldering in a piece of rod. This joint makes a good liquid collection point for an offset condenser design, because it covers the width of the liquid reflux. Like the through joint, the side-swipe is quite strong.

Sealing large containers

One of the most difficult jobs anyone can face is to provide a good, gas-tight seal for a lid on a large container like a boiler.

You can buy excellent "band" clamps with matching seals, just like those used for stainless steel couplings, and they offer a quick and effective solution, but are not available everywhere. What are the alternatives?

Home made seals or gaskets

The first step in sealing a pot is to make an effective gasket for the lid. You are very unlikely to find a ready-made seal just the right size or shape. It used to be that the only solution was to make a flat gasket out of paper, rubber or soft metal, or to resort to crude (but effective) measures like flour paste.

Today, Room Temperature Vulcanizing (RTV) silicone sealants are readily available, and they make the job very easy. These sealants are often intended for outdoor use, and may contain toxic chemicals. You want the kind marked “acceptable for food contact” or "aquarium grade", which is clear and not colored.

To make an RTV gasket for a pot, first clean the mating surfaces with acetone to remove all traces of grease or oil. Smear the top edge of the pot with petroleum jelly, and apply RTV liberally to the outer edge of the lid. Put the lid firmly on top of the pot and wait for the sealant to cure (24 hours is best). This makes an excellent, semi-permanent seal that remains attached to the lid. Of course, by choosing where you put the petroleum grease, you can make it stick to the pot, or make it so it comes free as a separate item. Since the sealant is rather weak by itself, we recommend leaving it permanently attached to a strong surface. If it ever becomes damaged, it can be removed with a razor blade.

Clamps

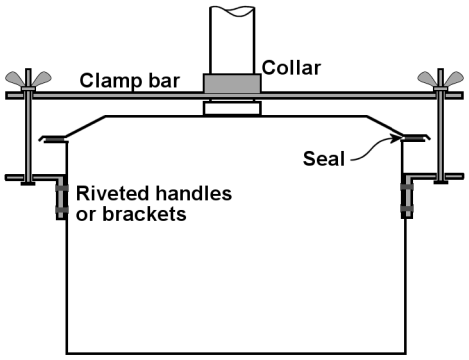


Fig. 7-10

Now that you have a gasket, the lid needs to be securely clamped to the pot, especially if it is also supporting a long top-heavy column, which could easily topple over.

You can rivet brackets onto the sides of the pot, and use these to secure clamps that hold the lid on, but you have to make sure that the rivets don't cause more problems than they solve. Solid rivets used to be common, and good kitchenware still uses them. Modern "pop" rivets will hold the brackets firmly in place, but are unfortunately hollow and not gas-tight. If you do choose to use them, plug the holes through the rivets with self-tapping screws.

If the pot comes with handles, then all your troubles are over! You can easily make the sort of clamp that is used to secure inspection hatches onto whiskey stills. This clamp is simply a bar (or shaped plate) that crosses over the top of the lid and which, when screwed down using the handles as anchor points, presses the lid down firmly and evenly onto the top of the pot. No handles? Rivet or braze on your own supporting handles or brackets!

Support

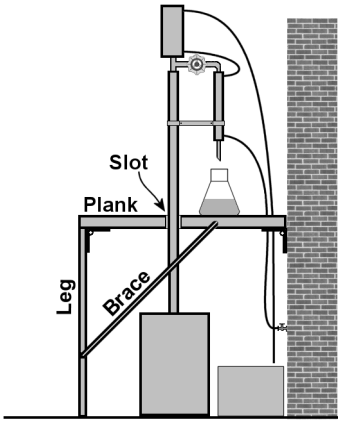


Fig. 7-11

Clamps hold pieces together, but how do you support the whole device? A boiler, a tall column, a still head and one or two condensers makes a top-heavy assembly, just asking to fall over. It should be supported at the top, or in the middle. The simplest way is with a bracket attached to a wall, but another method offers additional benefits.

Fasten one end of a wooden plank to a wall with a hinge, and fasten a drop-down leg to the other end. Cut a slot for the column partway through the middle of the plank, and you have a support that also creates a handy table! If the drop-down leg is properly braced with a cross-bar, you have a platform that is less wobbly than a conventional table, because it's fastened to a wall.

Heating elements

Heater control is important in operating a still to produce the best possible product. The greatest flexibility is obtained with fully variable control, like you get with a commercial hotplate or the electronic circuit detailed in Appendix 5. This will prove to be the most satisfactory method, but adequate control may be gained by clever use of heating elements.

Power control

Many people have tried many different methods of power control for electrical heating elements. Many of these methods violate electrical codes and international regulations, and will either attract attention from the power company or void your homeowner's insurance.

There are two fundamental approaches to power control:

- **Fully variable control.**
  For reasons spelled out in Chapter 4, light dimmers and motor speed controllers must not be used for heating element control. They are not designed for the power load of a heater, and generate a lot of electrical noise. If you want to have fully variable electronic control, then please use the circuit we have provided in Appendix 5, or purchase a compliant control box for a stove or commercial hotplate element. These circuits will not violate the new regulations, and are designed to handle the power of a heating element.

- **Stepwise control.**
  Boilers can be very effectively controlled if a few carefully-chosen values can be easily selected. One way of doing this, half-wave rectification, is an antiquated technique that definitely violates power regulations. The remaining methods include using multiple heating elements in various combinations, selecting between multiple voltages, or a combination of both techniques. Selecting multiple voltages is only possible in some countries, and having multiple voltages available in a single outlet creates a potential accident situation, so we do not recommend it.

Before we show how to design and build a reliable, multi-step control system, we need to discuss some electrical theory, so you can tailor your system to your needs.

Basic electricity

There are four key electrical relationships that are often used:

- If heating elements are connected in "parallel", so each has the full supply voltage across its terminals, the total power delivered will be the sum of each of the individual elements:
  $P_{TOTAL} = (P_1 + P_2)$

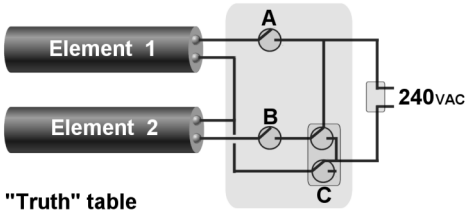
- If they are connected in "series", so current first flows through one and then the other, the total power delivered can be calculated from the relationship: **$1/P_{TOTAL} = 1/P_1 + 1/P_2$**
  $P_{TOTAL} = P_1P_2 / (P_1 + P_2)$

- Voltage, Current and Resistance are related by Ohm's famous Law:
  Voltage (volts) = Current (amps) x Resistance (ohms)
  V = I x R

- Power, voltage and current are related by this relationship:
  Power (watts) = Voltage (volts) x Current (amps)
  W = V x I

  From which, with Ohm's Law, we can derive two further relationships:
  $W = V^2/R$ and **$W = I^2R$**

Truth Tables



"Truth" table

A	B	C	Power
0	0	0	0
1	0	0	P_1
0	1	0	P_2
1	1	0	(P_1+P_2)
0	0	1	0
1	0	1	0
0	1	1	0
1	1	1	$P_1P_2 / (P_1+P_2)$

0 OFF
1 ON

Fig. 7-12

A "Truth Table" is a means of checking that you have considered the effects of all the possible ways in which switches can be set, and that no dangerous connections will be made. The usual way of denoting that a switch is "ON" is to use the number "1", and when it is "OFF" to use the number "0".

The Truth Table in Fig. 7-12 describes the outputs you get when the switches of that example circuit (which happens to be the one we talk about later, and show in Fig. 7-14) are set in all possible ways. You can check that all possibilities have been covered by considering that if each switch can be either ON (1) or OFF (0), two ways, then there must be 2 x 2 = 4 ways of setting two switches together, 2 x 2 x 2 = 8 ways for three switches, and so on.

Ways of controlling Power

Electrical services differ from country to country. Methods that work well with a 240V system will not work with 120V. Methods that work where both voltages are available are useless where only one voltage exists.

Most North American homes have both 120V and 240V available, although many, especially in areas served by gas, do not have any 240V circuits installed. Most of the rest of the world uses 240V exclusively. We have written three separate sections on stepwise control – one for people who have both 120V and 240V, one where only 120V power available, and one for those with only 240V.

If both 120V and 240V are available:

It is NEVER safe to run a 120V rated element on 240V, but it is always safe to plug a 240V rated element into 120V power, because its output is reduced to a quarter of its standard rating. So, if both 120V and 240V are available, then you can easily achieve two levels of control by operating **ONE** 240V element at two different voltages. Please remember that – **use only 240V rated heater elements with dual voltage control, and only one at a time!**

In North America, where both voltages are available, they have differently designed sockets so that devices cannot be accidentally plugged into the wrong voltage. In order to use both voltages for heater element control, you will have to build a short "jumper" cord that has a plug for one voltage on one end and the socket for the other voltage on the other. This cord must be made of heavy-duty materials and wire rated for 20 amperes. This kind of wire can be found at larger hardware and electrical supply stores. Connect a 240V plug to the element and make the jumper with a 240V receptacle for this, and a 120V plug at the other end. Use this jumper cord when using 120V power, and a standard 240V extension cord when using 240V. This will prevent any 120V appliance accidentally being plugged into 240V.

Most 240V power supplies are rated at either 15 or 20 amp, though larger circuits exist for stoves and clothes dryers. Each circuit capacity has its own design of plug and socket. You will have to determine the size of your circuit and buy the appropriate plug. Two common and useful sizes of 240V water heater elements are 3800W (15.8 amp) and 4500W (18.75amp), both of which require at least a 20-amp circuit.
3500W (14.6 amp) and smaller elements, all of which draw less current, are also available.

Using a 3800W element as an example, you will have 3800W available for rapid heat-up, and when the element is plugged into the 120V supply, the power will drop to 950W for reflux operation.

If only 120V is available:

Using a single power supply voltage gives you the opportunity to use two heater elements to safely achieve power level control.

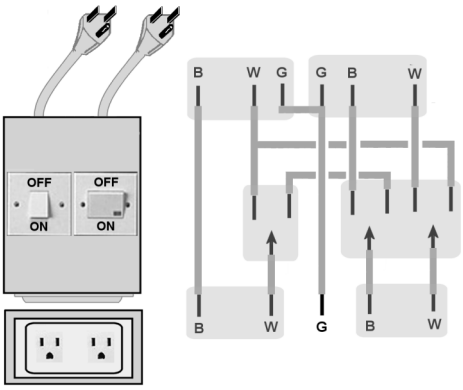
120V circuits are generally rated for 15 or 20 amperes, depending upon the wire gauge used. In North America, the largest 120V heating element available is 1500W, which draws 12.5 amperes. 1500W hotplates are readily available and inexpensive, and 1500W 120V water heater elements are available at most hardware stores.

If you are willing to accept the long heat-up time, you can certainly run a pot still with one 1500W element. With good attention to detail, you can probably also run a compound still at 1500W with good results. Obviously, having only one element and using only one heat level is the simplest possible design.

If you wish to heat up faster, you can install two separate 1500W elements in your boiler, **and plug them into separate circuits**. If you plug them into the same circuit, you WILL blow a fuse or breaker. Many rooms have several circuits available, but some do not. You may have to use an extension cord to bring a second circuit to your work space. Doing this will give you 3000W for rapid heat-up, but still leave you at 1500W minimum power.

Since heater elements draw large amounts of current, the circuit(s) used to feed them should not be feeding any other devices when the still is operating. In some of the possible arrangements, as small an additional load as a television set could trip the main breaker.

120V Control Box



Three levels of heat control can be achieved by building the box detailed in Fig. 7-13.
This box contains two outlets, one for each element, two switches and two cords for plugging into separate circuits. It allows you to operate one element for 1500W, two elements in parallel for 3000W, or both elements in series for 750W. This circuit is carefully designed to have no unsafe settings (make up a Truth Table and check for yourself!)

Fig. 7-13

120V Jumper Plug Box

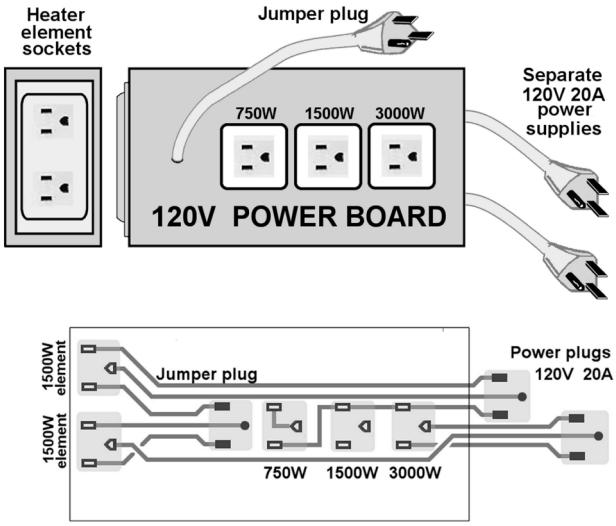


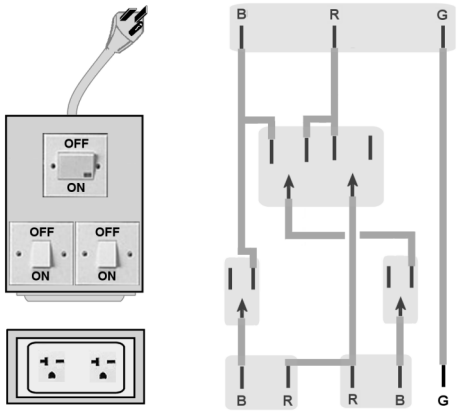
Fig. 7-14

Alternatively, as switches can be very expensive, you might prefer to do away with them altogether and make a Jumper Plug box instead. Not only is it cheaper, it is also easier to make, and the power levels can be clearly marked against each jumper socket.

Study the diagrams carefully, and if you have any doubts about your ability to build either of these devices, have a qualified friend make one for you. Also carefully mark it and keep the box in safe storage – it should not be used for **any** purpose but controlling your boiler heater elements.

If only 240V is available:

240V Control Box



If only 240V power is available, then you can use two elements and use them either independently or both together, either in parallel or in series.

A different type of control box is needed, as with 240V it is safe to plug into just one wall socket. The diagram shows two heater elements connected to a control box, which is in turn plugged into the 240V power supply wall socket. There are just three switches: a simple ON/OFF switch for each of the elements, switches A and B, and a DPDT switch to change from parallel to series operation, switch C.

Fig. 7-15

By using two different sized heater elements, you can achieve a range of different power settings. One useful design uses 1000W and 2000W elements. These give you the following selection of powers: **667W, 1000W, 2000W, and 3000W** off a 240V, 15 amp power supply. You might like to explore other combinations using the basic electrical relationships we gave above, but always remember to check the current by dividing the wattage by the supply voltage. Any combination larger than 3500W requires at least a 20 amp circuit.

Again, we've designed an alternative to switches, so you have a choice.

240V Jumper Plug Box

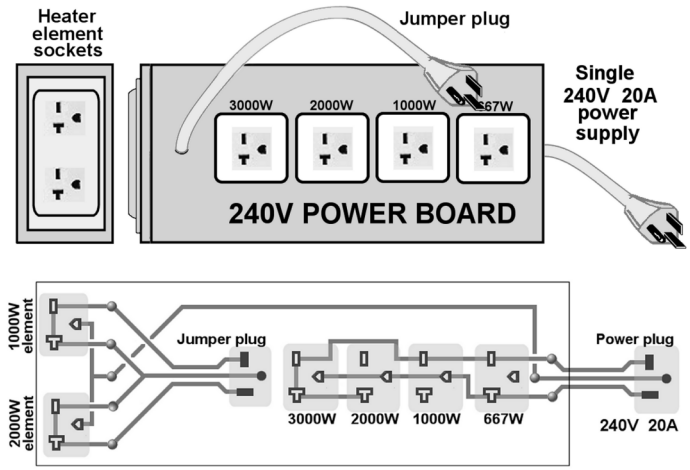


Fig. 7-16

Building Control Boxes

The box itself should preferably be made of insulating material, or firmly attached to the bare ground or earth wire. All wiring must be firmly connected and properly insulated. The wall socket is protected by a circuit breaker or fuse, but it can't hurt to provide extra protection by mounting two fast-blow fuses in the control box itself, one in each heater element line and rated for the maximum current to be drawn. (UK residents will have plugs that incorporate 10amp fuses, providing the same protection up to 2400W). The bare ground or earth line should also be attached to the boiler itself.

Switches

The style and selection of available switches and outlets varies greatly from country to country. The important point in selecting switches for your control box is that they should be able to handle the load. Select commercial or industrial grade switches rated for 20 amperes, and double check that the voltage rating is 240V or higher (most are actually rated at 600v), whether you are making the 120V control box or the 240V version. These are not standard household switches, but should be available in an electrical supply store or a large hardware store. All wire used should be at least 12 gauge, and 10 gauge would be a better choice.

We use two types of switches in the control boxes we have designed, and it might be useful to describe them briefly for those unfamiliar with such things.

SPST. This is short for "Single Pole Single Throw". This is used when you need to close or open a connection in a single line to another single line. It is a simple ON/OFF switch and has just 2 terminals.
SPDT. This is short for "Single Pole Double Throw", and is used when you need to close or open connections to either of two other lines. We've shown this latter type in Fig. 7-14, but SPST could be used instead. It has 3 terminals.
DPDT. This is short for "Double Pole Double Throw". In operation, it's exactly the same as if you has two SPDT switches joined together, and so has 6 terminals. This is used when such connections have to be made simultaneously, as leaving one closed when the other is open might lead to a dangerous configuration. These switches are used in both the 120V and 240V control boxes.

The 120V control box has two leads to plug into two 120V wall sockets, and the 240V control box has one lead to plug into one 240V wall socket. Both have two power sockets to supply power to the elements. The plug and sockets can either be mounted directly on the box, or connected to it by a length of 10 gauge flexible cable. Each heater element has its own plug, appropriate to the voltage being used, and all you have to do is ensure that it's plugged into the correct control box socket. Color coding the plugs and sockets, or even using two different designs of sockets will help keep things straight. If they are plugged into the wrong sockets, it isn't dangerous, but the intermediate power settings will not be what you expect, because switch A will control element B, and vice versa!

Since no heat is generated inside the box, it can be sealed tight, and does not need any ventilation holes.

Mark the element plugs, sockets and switches clearly and, if using switches, attach a copy of the truth table, indicating the power provided by each switch setting.

Soldering

Soldering is the normal method for connecting copper plumbing pipes. This is a very different thing than the sort of soldering done on electronic circuits with small soldering irons. Heavy duty soldering of large metal parts requires flame, and sometimes a lot of it.

You will need those safety goggles! Hot solder and corrosive fluxes can spatter alarming distances. Since you will be handling hot metal, a pair of good quality leather workshop or welding gloves is a very good idea. Metal stays very hot for a long time, and a piece that has been sitting for several minutes may still be hot enough to give you a nasty burn. The wet rag test works well – before touching anything that has been near a flame, touch it first with a wet rag. If it sizzles, then so would you if you touched it with your bare hand! Another good idea is to keep a pail of water in the workshop. When you have soldered a piece together, leave it to cool in air for a minute or so (sudden cooling of solder weakens the joint), then bring the whole thing to a temperature you can safely handle by plunging it into the water. This will be a good opportunity to give it a good scrub to remove excess flux.

To solder large items, you will need a hand-held butane or propane torch. For silver soldering and brazing, a set with a separate oxygen tank makes a MUCH hotter flame. You need solder and **flux**, and the **only** kind you should get is lead-free! Flux is a paste that you paint on the surfaces to be soldered. It dissolves the oxides that form a barrier between the solder and the metal. There will be a warning on the container, but it bears repeating here: treat plumbing soldering flux with respect! It is acidic and corrosive. Wash it off your work when you finish soldering, and wash yourself carefully if you get it on your skin.

A useful tool is an old fashioned soldering iron – a large block of copper that you heat up in the flame and which holds a great deal of heat. We didn't include this in the original list of tools as most jobs can be easily done without its help, but it can be very useful when soldering small parts. Don't even think of an electric soldering iron for electronic circuits. It can't generate nearly enough heat.

Solder is not capable of filling large gaps or voids. Pieces to be soldered should fit together snugly, so melted solder can flow into the joint by capillary action. Clean the surfaces to be joined with a wire brush and/or emery paper, then paint with plumbing soldering flux and assemble the joint. Heat until the solder melts on touching the hot metal. **NEVER** melt the solder directly in the flame. If the metal is hot enough to melt the solder, then the solder will run freely and be rapidly sucked in to coat and join the flux prepared surfaces. As the solder is being sucked in, play the flame on the part **towards** which the solder is flowing. This will help it along its way and ensure a sound joint. Afterwards, wire brush and wash the soldered joint thoroughly to remove all traces of flux that may remain.

There may be times when it is difficult to assemble parts so they hold together on their own as you solder them. This is especially true with small parts. In these cases, you can apply solder to the separate surfaces first (a process called **tinning**), then clamp or wire the parts together, and apply heat to re-melt the solder and create the joint.

Brazing (Silver Soldering)

Silver soldering and brazing are two techniques similar to soldering, but using materials that melt at a higher temperature and which have a much greater strength. Silver soldering uses solder containing significant amounts of silver. The term "brazing" comes from the use of brass alloys, but many different kinds of alloys are available for brazing. The temperature at which silver solder melts is much higher than ordinary solder, but lower than brazing alloys.

The temperatures used in silver soldering and brazing are so high that special heat-blocking glasses or safety goggles should be worn. There is enough infra-red energy in red-hot metal to damage your eyes.

Silver soldering is generally used where great strength is required in a joint (like the flange at the bottom of a column), and, like regular soldering, relies on capillary action to draw the solder into the joint. This means that parts to be joined need to fit tightly together. Silver soldering needs a different flux because of the much higher temperatures. A traditional silver soldering flux is borax, which requires skill to use. There are many fluxes available that are easier to use and quite effective, but many of these fluxes contain fluoric acids and must handled cautiously.

A propane flame is barely able to successfully silver solder small parts, and will not succeed with large components. You will need a MAPP gas torch, or a brazing set with an oxygen tank to generate the temperatures needed. Another useful technique is to conserve heat by building a small "hearth", which can be as simple as four bricks – two together form a base, and the other two on their long sides form walls enclosing a corner. Place the parts to be silver soldered in the corner and much of the heat you apply will build up, allowing the parts to reach the needed temperature. At the proper temperature, copper will be red hot, and brass will just be beginning to glow – within a few degrees of melting!

The "secret" of successful silver soldering is to make the surfaces to be joined as snug a fit as possible – the tighter the better. As with ordinary soldering, clean the surfaces first with a wire brush and emery paper, then paint with the flux. Now join the parts together tightly. If you can see a gap, chances are that it's too wide. After reaching the required temperature, the silver solder wire will melt when it touches the hot metal. If you've done everything right, then the molten solder will quickly disappear, sucked by capillary action between the surfaces to be joined. Once the job has cooled down, clean all the surfaces thoroughly to remove the last traces of flux.

Brazing uses similar techniques, but the alloys used do not become as thin when melted as solders. Brazing alloys have the advantage of being able to fill large gaps or even to be “built up” on surfaces. Brazing alloys are usually sold in thin, stiff rods, which are often coated with the appropriate flux. Brazed joints are very strong, and can also join dissimilar metals.

If you have any doubts whatsoever about your ability to handle objects at these high temperatures, do yourself a favor and find a skilled friend or a workshop to do the job for you.

Annealing

A torch is not only useful for soldering or brazing, but can also be very useful for **annealing** metals. Metals don't have a uniform structure – they're crystalline. If you bend a piece of metal back and forth until it fatigues and breaks, and look closely at the broken surfaces, you'll see the metal crystals exposed on the surface. You might need a magnifying glass, but they're there. When heated to a high enough temperature – usually red hot – these crystals tend to grow together and form larger crystals. The friction between the crystals creates much of the strength in many metals, so making the crystals larger makes the metals softer and easier to work. Hammering or bending the metal breaks the crystalline structure and causes metals to **work harden**.

Probably, the only metal you'll want to anneal is copper. Fortunately, annealing copper is extremely simple. All you need do is heat the copper to red heat and then let it cool. You can leave it to cool in the air, or quench it suddenly in a bucket of water, because copper doesn't temper like iron or steel. After annealing the copper will be much softer and easier to work, but if you keep bending a piece of copper or hammer it, then the large crystals will break up again and the metal will harden. You may find annealing necessary if you have trouble winding a copper tube to make a condenser coil. Too many attempts to correct bends will harden the metal, and the only answer then is to heat the hardened section until it is red hot and let it cool down. We mentioned rivets earlier when dealing with sealing the top of a large pot to make a boiler. Solid rivets are hammered into shape when applied to a job and so harden very quickly. It often pays to ensure that they are as soft as possible by annealing them before beginning a job.

A final tip for the Distiller

Scrubbers

We believe that metal scrubbers are the best packing material to use in a distillation column. However, people have more problems handling scrubbers than with any other part of their still! More often than not, scrubbers are taken straight out of the packet and pushed directly into the column. This is the easiest way to ensure that they're packed too tightly. Since they have a rough surface, the scrubbers tend to grip the sides of the column. Shoving them into a column is rather like trying to push an unwilling cat through a narrow hole – it tends to resist!

We've found that the best procedure is to pull the scrubber out into a loose cylinder, which should slip easily into the column. Remember, we want a lot of airspace inside the scrubber so vapor can get through easily. If you still have trouble with them, you can cut them into smaller pieces that will slip easily into the column without bunching up. After filling the column with scrubbers, you should be able to blow through it relatively easily. If you feel resistance, you have packed the column too tightly.

Another test for proper packing is the ability to get the scrubbers back out. In a properly packed column, the scrubbers will remain in their place, but can be easily pushed out with a large dowel or a pipe that fits inside of the column. This same dowel or pipe can be used to gently push scrubbers down into the column when packing it.

Another way of getting scrubbers out, especially with smaller diameter columns, is to fasten a fishhook on the end of a long wooden stick. Straighten out the hook, and the barb at the end will catch on the scrubber material, allowing you to pull it out.

Work safely!
Have fun!!

CHAPTER 8

THE SCIENCE BEHIND THE CURTAIN

Remember the scene in the movie "The Wizard of Oz" where the giant talking head insists that Dorothy and her stalwart crew should "Pay no attention to the man behind the curtain"? Well, many of the books and articles available on distillation take that same approach, telling you how to build a particular piece of gear or perform a specific distillation, but never explaining WHY you would want to do it that way.

In this chapter, like Toto, we pull aside the curtain and reveal the inner workings of the process. Here are all the reasons why we design and operate the way we recommend. These explanations would disrupt the flow of the book, so we have collected them together here. You can successfully accomplish anything written about in other chapters without reading this chapter, but we believe that you will enjoy it a lot more if you have read it. This chapter will give you many of tools you can use to design, diagnose and operate a wide variety of distilling devices for almost any purpose.

But be careful! Experience teaches that the more you dig into a treasure trove of information like this, the more fun you will have, the more ideas you will generate, and the more insight you will gain into this most interesting of hobbies.

Atoms and Molecules

You don't have to be a rocket scientist to understand how distillation works, but it does help to know a little about chemistry – the science of atoms and molecules. Everything is made up of atoms, and those atoms are joined together into millions of different kinds of **molecules**. Molecules are combinations of atoms, and each different combination forms a unique substance. If a molecule contains more than one type of atom, it is called a **compound**. We only have to concern ourselves with the properties of a few compounds (water, ethanol, and some congeners), so we can learn what we need to know quite quickly.

The molecules of compounds have their own characteristics, such as size, shape, mass, and electrical charge, which determine the properties of the substance. Painstaking research has determined the relative masses of all atoms, and hence of all molecules. Since atoms and molecules are so extremely tiny, their mass can't be meaningfully discussed in terms of grams or ounces; a new reference for mass was needed. Over the last 100 years, chemists have changed the standard of reference for the mass of an atom or molecule three times, as they gained more and more knowledge about how matter is constructed. The first standard of mass was Hydrogen, then Oxygen was selected, and finally in 1971, Carbon was settled on, and it remains the standard to this day.

The differences between these standards is in fact very small, usually found in the second or third decimal place. For example, in the Oxygen base system, water has a molecular mass of 18.016. In the Carbon system, the mass is 18.015.

For the purposes of calculating anything to do with distilling, rounding to the first decimal place is usually more than enough accuracy, so this really doesn't affect us. Here are the relative masses of some of the compounds you'll encounter when distilling ethyl alcohol:

water	18.016 atomic mass units,
methanol	32.042
ethanol	46.068
propanol	60.094
butanol	74.120

Moles and Mols

Knowing this scale makes relative measurements easy, because 18.0 grams of water has the same number of molecules as 46.1 grams of ethanol. Such quantities, assigning a practical unit of measurement instead of using "atomic units" is called a **mole**, and if using grams then 18.0 grams of water would be 1 gram mole, and 18.0 kilograms of water would be 1 kilogram mole. Of course, a kilogram mole of water would have 1000 times as many molecules as a gram mole, and if you used pounds for your measurements, then a "pound mole" would have yet another different number of molecules. The advantage of this system is that if you stick to any one practical unit of measurement, then 1 "whatever-it-is" mole of one substance will have exactly the same number of molecules as 1 "whatever-it-is" mole of another substance. If you measured out 18.0 pounds of water and 46.1 pounds of ethanol, you would have the same number of molecules of each compound. The unit of measurement used most frequently in chemistry is the gram. To save time, a "gram mole" is often referred to simply as a "**mol**", and this is what we'll mean from now on. Chemical engineers often use the kilogram mole, and this is distinguished by having a capital letter, **Mol**.

You must be very careful to use the correct molecular formula when calculating mols. For example, oxygen atoms have a mass of 16 atomic mass units, but 16 grams of oxygen is not a mol of oxygen gas, it's only half a mol. This is because oxygen normally exists in molecules containing of two oxygen atoms bound together, with a combined mass of 32 atomic mass units. So one mol of oxygen gas has a mass of 32 grams.

Molecular structures

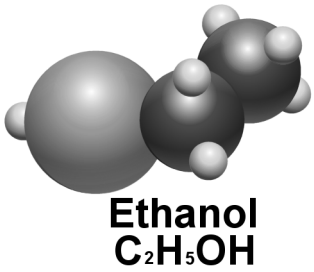


Fig. 8-1

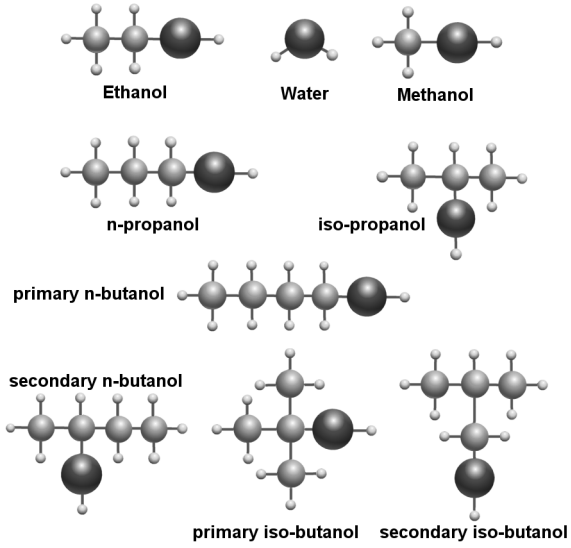


Fig. 8-2

The structures of the simple alcohols follow a simple pattern, which is easy to understand from a few pictures. The formulae for the first three alcohols are depicted pictorially in Fig. 8-2. Notice that there are two different pictures of propanol, and four for butanol. This is because there are more than one ways to arrange the molecules of some substances, and these different arrangements of atoms are called **isomers**. The structures shown here are simply two-dimensional representations of three-dimensional reality. We've attempted to improve on that in these other drawings of ethanol and water.

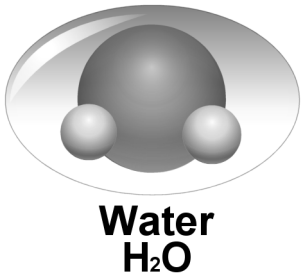


Fig. 8-3

You can see that all the alcohols follow a very simple progression dependent on the number of carbon atoms in their structure. Isomers of a compound have the same chemical properties, but their physical properties such as boiling point are affected by the different way they're physically put together. Have you noticed how much smaller the water molecule is than the others, and the way that the hydrogen atoms are naturally arranged at an angle to the oxygen atom? That will prove important later on!

Avogadro

Why all this attention to measuring by mols? The reason's really quite simple. A mol of water, weighing 18.016 grams contains the same number of molecules as one mol of ethanol, weighing 46.068 grams. Knowing the relative number of molecules greatly simplifies the calculations for chemical and physical phenomena. The actual number of molecules in a mol is named after the scientist who first proposed the concept, Lorenzo Romano Amedeo Carlo Avogadro, conte di Quarequa e di Cerreto (1776 - 1856). The number of molecules in a **mol** (remember, this is a **gram mole**)is defined as 6.0221367×10^{23} molecules, and this is known as Avogadro's number. This number is so huge that it's difficult to comprehend, so here are a few examples for perspective:

- A mol of standard soft drink cans would cover the surface of the earth to a depth of over 200 miles.
- A mol of unpopped popcorn kernels, spread across the United States of America, would bury the country over 9 miles deep.
- If you counted atoms at the rate of 10 million per second, it would take about 2 billion years to count the atoms in one mol.

Fortunately, we don't need to remember this number as it cancels out in all the equations we'll be using. However, knowing it helps to remind us just how incredibly small atoms and molecules are.

Volumes of Vapors

In addition to being very useful in calculating quantities for reactions (e.g. calculating how much sugar it takes to produce a certain quantity of alcohol in a fermentation), mols are extremely useful to distillers, because the **number** of molecules, not their size or weight, determines the volume of a vapor. This means that equal mols of vapor occupy the same volume. To be precise, one mol of a compound (in vapor form) occupies 22.4 liters at 0ºC and one atmosphere of pressure, conditions defined as **Standard Temperature and Pressure** (STP). Most of the vapors you'll be dealing with will be at atmospheric pressure, but not at 0ºC. Fortunately, you can calculate the volume they occupy at other temperatures and pressures by using the **Ideal Gas Law.** This law states that the pressure times the volume of a vapor, divided by it's temperature in degrees **Kelvin** (ºK=º + 273) is a constant. The equation is written PV=nRT, where n is the number of mols and R is the **Universal Gas Constant** (8.3144 Joules, or 1.9872 calories per mol degree Kelvin). Using this equation, you find that one mol of vapor will occupy 30 litres at standard atmospheric pressure when the temperature is 92.625 ºC (close enough to use 90 ºC for practical purposes).

Mol Fractions

The **mol fraction** is a very powerful tool when trying to understand what's going on in a mixture of compounds. When you're comparing the effect of the components of a mixture, the most useful way is to consider the proportions of molecules in the mix, which is done by measuring in mols, rather than conventional weights or volumes.

The mol fraction of a component of a mixture is the ratio of the number of mols of that component to the total number of mols of all the substances. As an example, the mol fraction of **Me** mols of pure ethanol mixed with **Mw** mols of pure water is **Me/(Me+Mw)**. It doesn't matter what the molecular weight of those compounds are, nor their density or any other measure – the only important factor is the numbers of molecules. We'll be using the mol fraction quite a bit from now on, so let's define some terms now.

In liquids, the mol fraction of a compound will be referred to by a capital **X** with a subscript to indicate which compound we're talking about. For example, **Xe** for liquid ethanol and **Xw** for liquid water.
So **Xe = Me/(Me+Mw)** and **Xw = Mw/(Me+Mw).**

In gases and vapors, the mol fraction of a compound will be referred to by a capital **Y** with a subscript to indicate which compound we're talking about. For example, **Ye** for ethanol vapor.
So **Ye = Me/(Me+Mw)** or **Yw = Mw/(Me+Mw).**

This may seem a bit confusing at first, but it helps you see at a glance whether we are discussing a liquid or a vapor.

Try this sum yourself: in any two compound mix, **Xa +Xb =1** and **Ya + Yb = 1**
This special relationship will be very useful in the following discussions.

Dalton's Law for Gases

In 1808, John Dalton stated that the total pressure in a system of gases is the same as the sum of the pressures of its components. This statement, made in his book "A New System of Chemical Philosophy", is now known as Dalton's Law, and amounts to saying that the total pressure is the sum of the pressures which each gas would exert **if it were confined alone in the volume occupied by the mixture**.

For example, we know that at 90°C a mol of water vapor occupies approximately 30 litres at atmospheric pressure. A mol of ethanol vapor at the same temperature would occupy the same volume at that temperature, so if both mols were to be combined at atmospheric pressure then the total volume would be 60 litres. But if we were now able to remove the ethanol vapor from the mix, but keep the same volume (the volume of the mixture), then the mol of water would exert only half an atmosphere pressure, and vice versa. In this case, the **"partial pressures"** of both the water and the ethanol are each half an atmosphere. You can arrive at the same conclusion another way by using the ideal gas equation, PV =nRT. If you double the volume of any vapor at a given temperature you halve the pressure.

Expressed in molecular terms used for vapors,
$\mathbf{Y_1}$ = mols of substance 1 in vapor/ (mols of substance 1 + mols of substance 2)
$\mathbf{Y_2}$ = mols of substance 2 in vapor/ (mols of substance 1 + mols of substance 2)
$\mathbf{P_{TOTAL}}$ = total pressure of the system
$\mathbf{P_1 = Y_1P_{TOTAL}}$ = vapor partial pressure of substance 1 in the mix
$\mathbf{P_2 = Y_2P_{TOTAL}}$ = vapor partial pressure of substance 2 in the mix

$$\mathbf{P_{TOTAL} = (P_1 + P_2) = (Y_1P_{TOTAL} + Y_2P_{TOTAL})}$$

In other words, from the molecular point of view, the ONLY important factor is the relative number of molecules present. The difference between the vapor pressure of a substance **on its own**, ($\mathbf{P_N^*}$), and its **partial** vapor pressure in a mixture ($\mathbf{X_NP_N^*}$ **for liquids**, or $\mathbf{Y_NP_{TOTAL}}$ **for vapors**) is very important.

Raoult's Law for Liquids (boiling of mixtures)

Later, Raoult extended this thinking to liquids. **Raoult's Law** states the vapor pressure contributed by each component of a mixture of liquids is the vapor pressure of that component multiplied by its mol fraction of the mixture.

This is because the mol fraction of a component governs the proportion of the liquid surface the component occupies, and the "share" of the surface occupied by a component controls its ability to escape and make pressure.

So, if
$\mathbf{X_1}$ = mols of substance 1 in liquid/ (mols of substance 1 + mols of substance 2)
$\mathbf{X_2}$ = mols of substance 2 in liquid/ (mols of substance 1 + mols of substance 2)
$\mathbf{P_1^*}$ = vapor pressure of substance 1 on its own
$\mathbf{P_2^*}$ = vapor pressure of substance 2 on its own
$\mathbf{P_1 = X_1P_1^*}$ = vapor pressure from substance 1 in the mix
$\mathbf{P_2 = X_2P_2^*}$ = vapor pressure from substance 2 in the mix

$$\mathbf{P_{TOTAL} = (P_1 + P_2) = (X_1P_1^* + X_2P_2^*)}$$

Saturated Vapor Pressures

If you raise the pressure or lower the temperature of a gas or vapor, the molecules become more crowded. As they move closer and closer together, the mutual attraction of the molecules eventually will overcome the thermal energy keeping them in a gaseous state, and droplets of liquid will begin to form in the vapor. A vapor that has reached this point is called **saturated**. A very common example of this process is fog – a suspension of tiny water droplets in air, which is a mixture of gases and water vapor.

A substance that is normally liquid or solid still has a vapor pressure, and for any specific temperature, there is a specific pressure at which it begins to condense into liquid – the **saturated vapor pressure**. The saturated vapor pressure is the maximum pressure that a substance's can exert at a given temperature. The experiment with the mercury barometer in chapter 2 was an illustration of saturated vapor pressure. The important point to remember about saturated vapors is that they contain the maximum possible number of molecules possible at that temperature. Whenever you work with vapors, you need know if they are saturated or not. Their behavior depends upon the answer to that question.

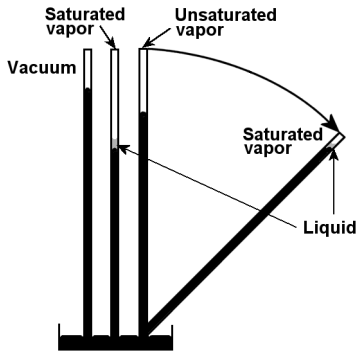


Fig. 8-4

This can easily be demonstrated with the same barometer experiment. Simply introduce a few drops of liquid, but not enough to saturate the space above the mercury, then **slowly** tilt the barometer so that the space gets smaller (slowly, to allow the heat generated by compression to escape and keep the temperature constant). At some point the liquid will start to condense out of the vapor, indicating that you have reached the saturated vapor pressure

In a distillation column, all the vapors are saturated throughout the system, and the temperature of the column at any point in the column is the boiling point of the mixture at that point. The boiling point of a substance is the point at which its saturated vapor pressure equals the pressure around it – in this case, atmospheric pressure. Just as saturated vapor forms above a boiling liquid, saturated vapor can condense back to a liquid at its boiling point This is the reason that reflux is so valuable.

Latent Heat of Vaporization (LHV)

The **Latent Heat of Vaporization** is the amount of energy a substance requires to transform from the liquid to the vapor state. If the liquid is being vaporized, this heat is absorbed; if it is condensing, the heat is released. The reverse, condensing the same amount of liquid, releases the same amount of energy. This is sometimes called the Latent Heat of Condensation (LHC), but as they are the same amounts, we'll just use LHV to mean both.

The following table lists the LHV for several substances you may encounter: water and the first four alcohols. The figures quoted are for standard atmospheric pressure.

Substance	**LHV** cal/gm	**LHV** kJ/mol	**LHV** Cal/mol
Water	539	40.639	9.7
Methyl alcohol	263	35.3	8.4
Ethyl alcohol	204	39.330	9.4
Propyl alcohol	160	40.3	9.6
Butyl alcohol	142	43.9	10.5

The figures expressing the LHV in calories per gram differ widely, so it's sometimes assumed that water needs far more heat to vaporize than any of the alcohols. This is true, on a **weight basis**, but if you look at the calories per mol, you can see that ethanol and water molecules require just about the same amount of energy to vaporize. To a first approximation, each requires 40 kJ to change from a liquid to a vapor, **and then occupies the same space as any other mol of vapor - 30 liters at 90ºC**.

If some of those units are confusing, the following list explains what each one is (the mol was defined earlier).

A **calorie** (cal) is a unit of **energy**, and is the amount of heat required to raise the temperature of 1 gram water through 1°C (it varies slightly with temperature)
A **Calorie** (Cal) is 1000 calories, and is the "Calorie" referred to when talking about food energy.
A **Joule** (J) is also a unit of **energy**, and is the amount of energy expended in one second by a current of 1 ampere flowing through a resistance of 1 ohm.
A **kiloJoule** (kJ) is 1000 Joules
From experiment, it's been determined that **1 calorie = 4.185 Joule**, so **1 Calorie = 4.185 kJ**

A **Watt** (W) is a unit of **power**, one Watt being defined as the **rate** at which energy is expended by a current of 1 ampere flowing through a resistance of 1 ohm.
So **1 Watt = 1 Joule/sec**.

Interesting and Useful Deduction
Since 1 kW = 1kJ/sec, a 1 kW heater will give out 60kJ of energy every minute, so 60/40 = 1.5 mols of either water or ethanol, **or any mixture of both**, will be vaporized every minute to give 45 liters of vapor at normal atmospheric pressure. This is a **very** useful thing to know!

Logarithms

A short note here for those who may not have used logarithms before, or are just a bit rusty.

If a number, a, is expressed as a power of another number, b (if $a = b^n$), then 'n' is said to be the logarithm of a to base b, written $\log_b a$.
'Common' logarithms are to base 10.
In mathematics and the sciences, 'natural' or 'Napierian' logarithms are often used as they can simplify calculations. These are logarithms to a base 'e', a number derived from the sum of the infinite series:
e = 1 + 1/1 + 1/1x2 + 1/1x2x3 + 1/1x2x3x4 + …… etc
and e = 2.71828…….
Such a number may not appear at first sight to simplify anything! However, as this is not a book on maths, please just accept our word for it that it does.
Many hand-held calculators have buttons for both 'common' and 'natural' logarithms, marked LOG (base 10) and LN (base e) respectively. The relationship between the two types of logarithm is
LOG(X) = 0.4343 x LN(X)

Clausius-Clapeyron Equation

We've discussed vapor pressures, and how you can calculate it if you know the number of mols, the volume and the temperature of the vapor. It would be tiresome to have to do this calculation every time you wanted to know what the partial pressure of a substance is at some temperature. The following equation, easily programmed into a spreadsheet, will give a value directly, allowing us to easily determine how a liquid/vapor or solid/vapor system behaves. The Clausius-Clapeyron Equation is:

$$\mathbf{\log_e(P/P_0) = H(1/T_0 - 1/T) / R}$$

where:
P is the vapor pressure at temperature **T** in degrees Kelvin (**°K = 273 + °C**)
$\mathbf{P_0}$ is a known vapor pressure at temperature $\mathbf{T_0}$ **°K**
H is the LHV if the substance is a liquid or the enthalpy of sublimation if it's a solid
R is the Universal Gas Constant (**R = 8.3144 J/mol.K, or 8.3144×10^{-3} kJ/mol.K, or 1.9872 cal/mol.K)**

All you have to do when using this equation is take care that the units you're using are consistent with each other.
For example, we know that the vapor pressure of water is 1000 mbar at its boiling point of 100ºC
The vapor pressure at 78.32ºC (the boiling point of ethanol) is $\mathbf{P_{78.32}}$ in the following sum:

$$\mathbf{\log_e(1000/P_{78.32}) = 40.639\ (1/373 - 1/351.32) / 8.3144 \times 10^{-3}}$$

$$\mathbf{P_{78.32} = 1000/2.245 = 445\ mbar}$$

Antoine Equation

The Clausius-Clapeyron equation is a very useful tool, but to use it, you need to know the vapour pressure $\mathbf{P_0}$ at a temperature $\mathbf{T_0}$, and the LHV of that substance **H**. For our purposes, this is not as difficult as it seems, because $\mathbf{P_0}$ is 760mm.Hg (1000 mbar) at the boiling point $\mathbf{T_0}$ of the substance, and the LHV is in the table above.

There are several other methods of calculating vapor pressure. One of the easiest to use is the Antoine equation, and it has some advantages. It is more accurate than the Clausius-Clapeyron equation over specific temperature ranges and it doesn't require you to look up the vapor pressure or LHV, because these are incorporated into parameters for each substance. The Antoine equation is:

$$\mathbf{Log_{10}\ P = A - B / (T + C)}$$

Where P is the vapor pressure and T is the temperature, and A, B and C are parameters for the particular substance.

If you'd like to try using this simple equation, here are the parameters for water and the first two alcohols. These parameters require that temperature be in degrees **Celsius**.

Substance	Temp. Range °C	A	B	C
Water	1 - 100	8.190447	1730.630	233.426
Methyl alcohol	15 - 84	8.205910	1582.271	239.726
Ethyl alcohol	20 - 93	8.236515	1592.864	226.184

When you use the Antoine equation to find the vapor pressure of water at the boiling point of pure ethanol, 78.32 ºC, the result is **436 mbar,** which is closer to the actual measured pressure.

The difference is that the Clausius-Clapeyron equation is derived from the theoretical behavior of a "**perfect gas**", while the Antoine equation's parameters are based on careful measurements and experimentation. Since the results of the two equations fall within 2% of one another, you can use either one quite comfortably for practical matters.

Equilibrium Curves

You now have enough information to work out what happens when you heat a mix of ethanol and water and send the vapor through a reflux column, by combining Raoult's and Dalton's laws with either the Clausius-Clapeyron or the Antoine equations. The following demonstration uses the Clausius-Clapeyron equation.

We know from Raoult that $\mathbf{P_{TOTAL} = P_1 + P_2 = X_1P_1^* + X_2P_2^*}$

And we know from mol fractions that $\mathbf{X_1 + X_2 = 1}$	so	$\mathbf{X_1 = (P_{TOTAL} - P_2^*) / (P_1^* - P_2^*)}$	**Eqn 1**
As $\mathbf{P_1 = X_1P^*}$ and also (from Dalton) $\mathbf{P_1 = Y_1P_{TOTAL}}$	so	$\mathbf{Y_1 = X_1P^* / P_{TOTAL}}$	**Eqn 2**
We can calculate $\mathbf{P_1^*}$ and $\mathbf{P_2^*}$ from Clausius-Clapeyron		$\mathbf{\log_e(Pn^*/P_0) = H(1/T_0 - 1/T) / R}$	**Eqn 3**

$\mathbf{P_0}$ and $\mathbf{T_0}$ are the pressures and temperatures of the pure liquids at their boiling points, something we already know for water and ethanol, and $\mathbf{P_{TOTAL}}$ is atmospheric pressure

These results show why it's important to keep the pressure constant, and also indicate what happens if you operate at higher or lower pressures. Equation 2 shows that raising the pressure lowers the yield of vapor, and that lowering the pressure (eg. vacuum distillation) increases it. We've seen many designs for stills that operate under high pressure in the hope that this will increase the yield. Not only is that practice dangerous, the equations show that it's based on ignorance.

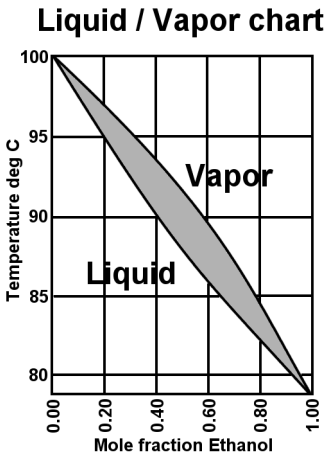


Fig. 8-5

These three equations, plotted for ethanol mol fractions ranging from 0 to 1 and temperatures from 78.5ºC to 100ºC, result in the following constant pressure plot. The lower curve is called the **bubble line**, because it shows the temperatures at which the mix begins to boil, and the upper curve is called the **dew line**, because it indicates the temperature at which vapor first condenses. Anything below the bubble line is liquid, and anything above the dew line is vapor. The area enclosed by the two curves represents points where liquid and vapor are in dynamic equilibrium.

This plot is extremely useful Because it lets you graphically plot the progress of a distillation. If, for example, you start with 11% ethanol in the boiler, the first vapor to come off contains 23% ethanol. If this is condensed and re-boiled, the resulting vapor would contain 40% ethanol, and so on up to the maximum possible concentration of around 96% ethanol. Ethanol has a maximum concentration lower than 100% because it forms an **azeotropic** mixture with water. We used charts like this in Chapter 2 to illustrate what happens with repeated distillations.

The stages in this plot are called **theoretical plates**. This name comes from the plates or trays inserted in large commercial distillation towers to facilitate the evaporation and condensation process. These plates serve the same purpose as the packing used in small-scale columns. On the diagram, a theoretical plate is marked out by going vertically from the bubble line to the dew line, and then horizontally back to the bubble line.

In the example starting at 11%, 7 plates are needed to reach the top concentration, and the first plate is the boiler itself. If a packed column is 100 cm long, and the temperature levels out at the boiling point of ethanol 60 cm up, then the **Height Equivalent Theoretical Plate** (HETP) for this packing would be 60/6 = 10 cm. If you are interested in calculating the number of theoretical plates needed in a column, you should use the McCabe-Thiele equations, and we use a simplified form of these in a moment when discussing Reflux. On the internet, Tony Ackland's website (see Appendix 8) gives an excellent description of them. Tony is a chemical engineer in New Zealand with a great interest in the hobby of distillation.

To save you the trouble of plotting this equilibrium diagram, and to calculate how the mol fraction of ethanol relates to percentage by volume, the following tables give the results of the calculations. Please remember that the equations relate to "perfect" gases, and don't exactly match the results you'd get with a very sensitive thermometer and controlled laboratory conditions. However, you should find them very useful in all practical situations.

Deg C	Xl	Yl	N%		Deg C	Xl	Yl	N%
100.2	0.00	0.00	0.00		89.5	0.39	0.59	67.81
100.0	0.01	0.01	1.93		89.0	0.41	0.61	69.81
99.5	0.02	0.04	6.60		88.5	0.44	0.63	71.76
99.0	0.04	0.08	11.05		88.0	0.46	0.65	73.63
98.5	0.05	0.11	15.29		87.5	0.48	0.67	75.45
98.0	0.07	0.14	19.34		87.0	0.51	0.70	77.21
97.5	0.08	0.17	23.21		86.5	0.53	0.72	78.91
97.0	0.10	0.20	26.92		86.0	0.56	0.74	80.56
96.5	0.12	0.23	30.47		85.5	0.58	0.76	82.16
96.0	0.14	0.25	33.87		85.0	0.61	0.78	83.71
95.5	0.15	0.28	37.13		84.5	0.64	0.80	85.21
95.0	0.17	0.31	40.26		84.0	0.67	0.82	86.67
94.5	0.19	0.34	43.26		83.5	0.69	0.83	88.09
94.0	0.21	0.36	46.15		83.0	0.72	0.85	89.46
93.5	0.23	0.39	48.93		82.5	0.75	0.87	90.80
93.0	0.25	0.42	51.60		82.0	0.78	0.89	92.09
92.5	0.27	0.44	54.18		81.5	0.81	0.91	93.35
92.0	0.29	0.47	56.66		81.0	0.84	0.92	94.58
91.5	0.31	0.49	59.05		80.5	0.87	0.94	95.77
91.0	0.33	0.52	61.36		80.0	0.91	0.96	96.92
90.5	0.35	0.54	63.58		79.5	0.94	0.97	98.05
90.0	0.37	0.56	65.73		79.0	0.97	0.99	99.15
89.5	0.39	0.59	67.81		78.6	1.00	1.00	100.0

Fig. 8-6

Note too that the figures for mol fractions and percentage ethanol for temperatures below 80°C on this chart are those derived from theory. In real life, ethanol is 'difficult' as it exhibits a phenomenon called azeotropism, and the figures depart from those that simple theory would predict. So, what is azeotropism?

Azeotropes

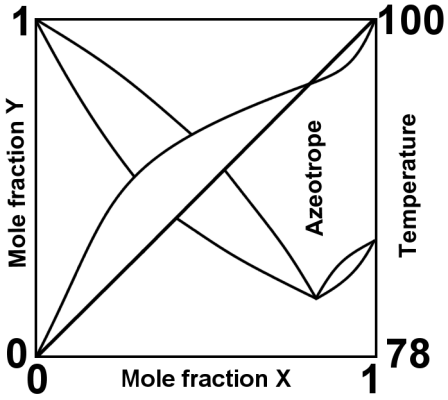


Fig. 8-7

An **azeotropic** mixture is one whose boiling point is lower than that of any other combination of the compounds in the mixture. This is caused by the attraction between the molecules of the mixture, and is not predicted by the ideal gas laws. When an azeotropic mixture vaporizes, the vapor has exactly the same concentrations as the liquid, which is why you cannot form a more concentrated mixture by distilling.

On the left, we've attempted to combine two graphs into one to explain the phenomenon.

The first graph plots the mol fraction **Y** of ethanol vapor you get from a mixture with mol fraction **X** of ethanol. This is the curved line running from bottom left to top right. Where it crosses the straight line joining the two ends, the mol fraction of ethanol in the vapor is the same as the mol fraction of ethanol in the liquid. Below that point the vapor is always **richer** in ethanol than the liquid it came from, and above that point the vapor is always **leaner** in ethanol than the liquid it came from. This is the azeotropic point.

The second graph plots the values of **X** and **Y** for all the temperatures from 78ºC to 100ºC. It is the pair of curved lines running down from the top left. These lines meet again before the temperature reaches 78°C, then run up and to the right. They finally meet again when **X = 1**. The lowest point of this graph matches the crossover point of the first graph, when the mol fraction of ethanol in the liquid equals the mol fraction in the vapor. These are the Azeotropic points and $\mathbf{X_A = Y_A}$. This shows why you can't get a higher percentage if ethanol than around 96% by distillation alone - at atmospheric pressure. Significantly lowering the pressure in the still (vacuum distillation) will produce a higher strength product, but is not worth the cost and danger.

Reflux

Why have reflux? Why is it important? After all, we've shown that repeated distillations will produce increasing concentrations of volatile compounds, and you can achieve this in a simple pot still by distilling the product several times. Eventually, you'll end up with a product containing a very high concentration of all the most volatile components of our original brew.

The crux of the issue is that little word "all". You will end up with all the ethanol, all the propanol, all the butanol, etc, etc. This may be desirable if making whiskey or brandy, and judicious selection of the "heads" and the "tails" of the distillate will ensure that just enough of these compounds are included in the final product. However, raw whiskey tastes awful and has to go through many stages of maturation over a very long time before it's palatable.

Refluxing means feeding a condensed product back to the still for re-distillation, so repeated pot distilling is a form of "manual" refluxing. However, it is very inefficient process and the losses as you discard enough heads and tails
to isolate pure ethanol mean that you end up with very, very little product after a great deal of time and effort.

We've already seen that a reflux column can do this for us, and that a compound column with imposed reflux can separate efficiently enough to allow collection of individual compounds. But what amount of reflux should be imposed, and why? Once again, theory and calculation can provide the answer.

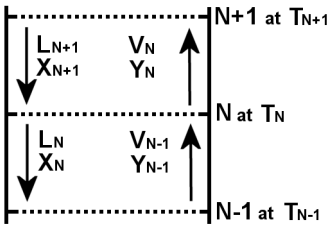


Fig. 8-8

Let's start by looking at a single stage of distillation, one "plate". Figure 8-08 shows a plate with the one above it and the one below it.

This plate, which we'll call "**plate N**", is at temperature $\mathbf{T_N}$
It's holding liquid that's boiling and giving off a quantity of vapor $\mathbf{V_N}$, and the mol fraction of its most volatile component is $\mathbf{Y_N}$

Having boiled at $\mathbf{T_N}$, the excess liquid drips down to the lower plate, "**plate N-1**". The quantity of this liquid is $\mathbf{L_N}$ and the mol fraction of that volatile component is $\mathbf{X_N}$

At the same time, **plate N** is receiving a quantity $\mathbf{V_{N-1}}$ of vapor from the lower **plate N-1** with mol fraction $\mathbf{Y_{N-1}}$. This vapor is at $\mathbf{T_{N-1}}$ so it condenses on the cooler **plate N** because $\mathbf{T_N}$ is lower than $\mathbf{T_{N-1}}$

To complete the picture, **plate N** is also receiving liquid from the upper **plate N+1**. The quantity is $\mathbf{L_{N+1}}$ and its mol fraction is $\mathbf{X_{N+1}}$. This liquid, at temperature $\mathbf{T_{N+1}}$, starts to boil when it hits **plate N** because $\mathbf{T_{N+1}}$ is lower than $\mathbf{T_N}$

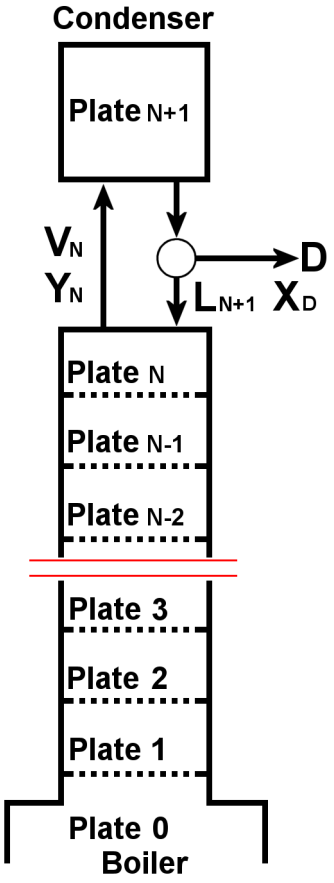


Fig. 8-9

Since a quantity of liquid **L** with mol fraction **X** of a component contains **LX** mols of that component, the **balance equation** at **plate N** for the volatile component we're considering is:

Total mols received = Total mols lost
$V_{N-1}Y_{N-1} + L_{N+1}X_{N+1} = V_NY_N + L_NX_N$

This equation will apply at every point in the column if it's in balance (ie. in equilibrium). You just have to change the suffixes to match the plate number.

The condenser on top of the compound column is considered the top plate, and the boiler is the bottom plate. The liquid returned to the column from the top condenser is a special case of 'imposed' reflux, unlike the 'natural' reflux generated inside the column. We've therefore set it apart by identifying the final mol fraction of the liquid coming from the condenser as X_D . So, if X_D is the final mol fraction of the liquid condensed at the top of the column, and L_{N+1} is the reflux, then

$X_{N+1} = X_D$

The balance equation for plate N may be written:
$V_{N-1}Y_{N-1} + L_{N+1}X_D = V_NY_N + L_NX_N$

You can consider the whole column and as one block unit.
Material entering this block $= V_0Y_0 + L_{N+1}X_{N+1}$
Material leaving the block is $= V_NY_N + L_1X_1$

Which means that $V_0Y_0 + L_{N+1}X_D = V_NY_N + L_NX_N$

But $V_NY_N = L_{N+1} + DX_D$ (consider what enters and leaves the condenser).

So $V_0Y_0 = L_1X_1 + DX_D$

This describes the balance of the column as a whole.

Constant Molal Overflow

In general, values of V and L vary from stage to stage and an LHV balance for each stage is required to calculate these figures. However,
IF the heat losses are negligible,
AND there is negligible heat of mixing,
AND the LHV values for the components are almost equal,
THEN a condition termed "Constant Molal Overflow" exists and the following simplifications can be made:

$\mathbf{L_{N-1} = L_N = L_{N+1}}$ = … etc
$\mathbf{V_{N-1} = V_N = V_{N+1}}$ = … etc

Assuming constant molal overflow, the balance equation for the whole column $\mathbf{V_0Y_0 = L_1X_1 + DX_D}$ becomes

$\mathbf{VY_0 = LX_1 + DX_D}$
Where **V** = vapor from top of column
L = reflux
D = top product

Dividing throughout by **V,** you get
$\mathbf{Y_0 = L/V * X_1 + D/V * X_D}$

At this point, we must stop and define a term: **the reflux ratio.**

Reflux Ratio

In this book, we define the **reflux ratio** as the ratio of the final product returned to the column to the total delivered to the condenser, and product ratio as the ratio of the product collected to the total delivered.

So the reflux ratio (**F**), equals L/V, and the product ratio (**1-F)**, is D/V.

If you collect all the condensate, returning nothing, then F = 0. If you return everything (operating under "full reflux") then F = 1.

The balance equation becomes $\mathbf{Y_0 = F * X_1 + (F-1) * X_D}$

Since our aim is to get as much of the volatile component as possible, let's set $\mathbf{X_D = 1.}$
The equation reduces to $\mathbf{Y_0 = F * X_1 + (F-1)}$
In this equation $\mathbf{Y_0}$ is the mol fraction of volatile component in the vapor leaving the boiler and $\mathbf{X_1}$ is the mol fraction in the liquid being returned to the boiler. Our aim is to get **all** of the volatile component, so $\mathbf{X_1}$ = 0.

We end up after all this with the simple relationship:

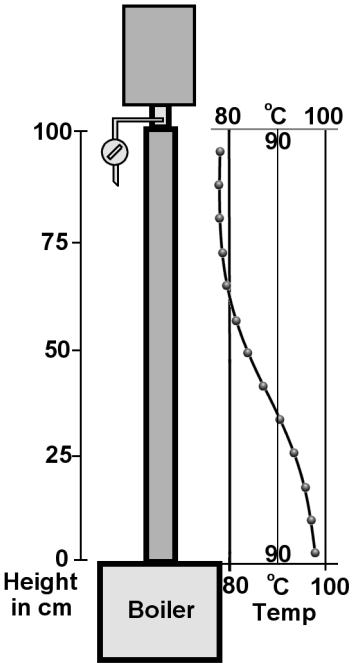
$\mathbf{F = 1 - Y_0}$

This equation shows that when the concentration of the volatile component in the boiler is very low, producing vapor with a very small $\mathbf{Y_0}$, **F** needs to be very close to **1** in order to achieve our aim (which means that you'd get hardly any product **D)**. The higher the concentration in the boiler, the smaller **F** can be, meaning you need less reflux.

This is the reason you need reflux in a column in order to get a pure product. A reflux column on its own will provide some of the total reflux required, but not enough to achieve complete separation. In practice, a reflux ratios in the range of 8/10 to 9/10 give good results with a column containing around 80cm of packing.

The simple equation for reflux ratio also shows why it makes sense to do a quick pot distillation before embarking on rectification (fractional distillation). This increases the concentration of volatiles in the boiler, greatly reducing the time needed for rectification. However, it also shows that towards the end of rectification, when the volatile concentration in the boiler is low and you're trying to squeeze the last traces of volatiles out of the mix, you must increase the reflux ratio to maintain quality.

Some important lessons can be learned from these calculations. Remember that they assume that the pressure is constant throughout the column, and that there are constant molal overflow conditions. One of the conditions for constant molal overflow is that heat losses are negligible. So, if you want your still to be as efficient as possible, **the column must be insulated very well**.



Fig, 8-10

Using packing rather than physical trays or plates, the spacing of the theoretical plates varies with the position in the column, and as the distillation proceeds. You can check this yourself by measuring the temperature at several points in column when it's in equilibrium. Figure 8-10 is a plot of the temperatures we found when distilling a mixture of ethanol and water. It shows that the temperature falls as you go up the column, but that near the top it levels out at a constant value.

The obvious conclusion is that the top section of the column is filled with ethanol vapor at its highest molar concentration, because the temperature is very close to the boiling point of 95% ethanol. You could argue that the whole section is acting as one "plate", but this is probably too simple. This plateau quickly disappeared when we reduced the reflux ratio, and the product started to smell like moonshine.

The calculations we've done using theoretical plates are valid and well proven, and the overall conclusions apply equally well to packing, as long as the packing has an adequate surface/volume ratio. Good packing material is the key to successful separation.

Packing

Packing material is hotly debated in amateur distilling circles, with questions like these constantly coming up:
"Should I use marbles? I've heard of Raschig rings - but what are they, and are they suitable for my equipment? I've heard that pot scrubbers make the best packing for small stills - but that sound silly"

This subject is a hotbed of opinions and, quite frankly, a lot of half-baked ideas. We need to go back to first principles to try and shed some light on this contentious subject.

Most people just don't understand how a plate in a large column operates. This is made worse by the fact that many of the equations used to describe the process use the term "theoretical plate". This leads to the notion that all you need to do is put flat plates in the right places inside a column, and the job will be done.

A plate in a large column is **not** just a flat plate. It's a trap to collect and hold reflux coming down the column, keeping it in intimate contact with the vapor rising up the column, so the equilibration processes can occur. Plates in big commercial columns have many different designs. One simple one is to stud each tray with numerous "bubble caps" that look like small metal mushrooms. Rising vapor enters the hollow stems of these "mushrooms" and then bubbles through the liquid because the outer lips of the caps are submerged in the liquid held in the tray.

The resulting foam of bubbles provides the large surface area needed for equilibrium exchange. If another way could be found to provide this large contact area between liquid and vapor then trays wouldn't be needed at all!

Bubble caps and foam are clearly out of the question for small columns, so the large liquid/vapor interface area is provided not by a foam of bubbles, but by the surface area of materials put in the column – the packing. The liquid reflux coats the large surface area of this material is held in intimate contact with the vapor. **The greater the surface area, the better the interaction between liquid and vapor.**

We can't use any material with a large surface area, because vapor and liquid have to flow freely through the column. Sand has a large surface area for a given volume, but the particles are so small that liquid would fill the tiny gaps between the grains and no vapor could pass through. Pebbles would permit vapor to rise as the gaps would be much larger, but the surface area would be very much less. We have to find the best compromise between the volume occupied by the packing material, the surface area available, and the space left for vapor and liquid to flow up and down. Let's examine a few frequently mentioned packing materials.

Marbles

Marbles are often touted as a packing material, but are actually one of the worst available, because the sphere is the solid with the lowest possible surface area. The surface area of a spherical marble is πD^2, where **D =diameter**, π is the Greek letter (Pi) representing the ratio of the circumference of a circle to its diameter, and the **volume is $\pi D^3 / 6$.**

Suppose we can pack **N** marbles into our column, and that this column has a volume **V**. The total surface area of all these marbles is $N\pi D^2$ and their combined volume is $N\pi D^3 / 6$.

The surface area these marbles can offer in the packed volume V of our column is the factor we're interested in, so let's define this term: the surface/volume ratio.

Z = surface area of packing / volume of column occupied by packing.

Note: it's the volume of the column that's packed, **not** the volume of the packing material itself, because we're interested in the free space left for vapor and liquid flow.

For marbles, $Z = N\pi D^2 / V$

We know **D**, and we know **V**, so all we need to know is **N**.

This may seem simple. After all, if we know the size of each marble it should be easy to calculate how many can occupy a volume **V**, shouldn't it? Unfortunately, the answer is no!

The problem of random particle packing has been fascinating mathematicians since ancient times, and is still not completely understood. Experiments have even been done on the space shuttle to study how packing is affected by micro-gravity. Given enough shaking, spheres will always settle to a maximum density, a state known as random close packing (**RCP**), and this density will be about **0.64** (volume of spheres / volume they occupy).

However, if you carefully stack the spheres, you can improve on this. This was predicted by Johannes Kepler (1571-1630), who calculated that a packing density of $\pi / 18^{1/2} = 0.74$ could be achieved. As a demonstration of how difficult these mathematical problems are, it wasn't until 2000 that Sal Torquato, a professor at Princeton University proved this to be true!

We should apply the RCP factor of 0.64 to calculate N (it is extremely difficult to achieve 0.74), but because the average marble's diameter is relatively close to the diameter of a small column and this causes interference, we will round that down to 0.6.

The total volume of marbles is $\mathbf{N\pi D^3 / 6 = 0.6V}$, and $\mathbf{Z = N\pi D^2 / V = 3.6 / D}$

For marbles with $\mathbf{D = 1}$, then $\mathbf{Z = 3.6}$. If you use marbles or ball bearings only 0.5cm diameter, then Z increases to 7.2

Cylinders

Cylinders have more surface area per unit of volume than spheres. For convenience, (and because it's a common ratio in commercial packing materials), we'll assume that our cylinders are twice as long as they're wide, so if the **diameter is d** then the **length is 2d**.

Also, to make the argument easier to follow, we'll make each cylinder out of one of the marbles. Doing the calculations, the width of each cylinder is $\mathbf{1/3^{1/3}}$ times the diameter of the marble, or $\mathbf{d = D / 3^{1/3} = 0.69336D}$. More math shows that the surface of our cylinder has an area of $\mathbf{2.5\pi d^2 = 3.776cm^2}$, an improvement over the marble which had a surface area of $\mathbf{\pi = 3.1416\ cm^2}$.

The volume of each cylinder is $\mathbf{\pi d^3 / 2}$, and assuming the same RCP factor of 0.6 (it is actually better with cylinders, but we're being conservative), $\mathbf{N\pi d^3 / 2 = 0.6V}$, so $\mathbf{N\pi d^2 / 0.6V = 2 / d}$

Thus the ratio $\mathbf{Z = 2.5N\pi d^2 / 0.6V = 5 / d = 5 * 3^{1/3} / D = 7.2 / D}$ (the marble was 3.6 / D)

The ratio **Z** for the cylinder is twice that of the marble with the same volume!

Raschig rings

A Raschig ring is a thin-walled hollow cylinder, and is a common industrial packing material.

The RCP is exactly the same as a solid cylinder, but we've doubled the surface area. So **Z = 14.4 / D** and thus Raschig rings are at least 4 times as effective as marbles, volume for volume ("at least", because the RCP for solid or hollow cylinders is greater than for marbles).

Metal Pot scrubbers

Let's do the same thing as we did with cylinders, make our pot scrubber from the material of one marble.
Pot scrubbers are made from long filaments with a rectangular cross section. Typically, the **width of a filament is 0.1cm and the thickness 0.001cm**, though this varies with different brands. We've done our calculations using these sizes because that's the type we use. You can do your own measurements and calculations with your scrubbers, but the conclusions will be much the same.

Let the width of the cross section of a filament be **W** and the height **H**. Let the length be **L**, and to simplify the math, imagine we can make one long filament out of our marble.

The volume of the filament will be **W * H * L**, and it will be the same as the marble it came from.

Since the 1cm marble has a volume of $\pi / 6$,

$\mathbf{0.001 * 0.1 * L = \pi / 6}$

So $\mathbf{L = 10000\pi / 6 = 5236\ cm\ (172\ feet)}$

Amazing, isn't it? Over 52 meters (57 yards) from one small marble!

The surface area is $\mathbf{S = 2 * 0.1 * 5236 = 1047.2cm^2}$ **(1.1 square feet)** (multiply by 2 because it has two sides).

RCP does not apply to a filament, because it is not a solid object like a marble, so we have to make some real-world measurements. We took a quantity of steel scrubber material we'd used in a column (and which had been stretched out so it didn't resist the flow of vapor and liquid), and measured it. Its volume was 464 ml (20 cu. In.) and it weighed 71 grams (2.5 ounces.

The density of steel is 8 gm/ml, so a 1cm steel marble weighs $8\pi / 6 = 4.19$ gram. The 71 grams of scrubber material is equivalent to 17 marbles. The surface area of a scrubber filament made from one marble is **1047.2cm^2**, so the total surface of the scrubber is **17 * 1047.2cm^2**, in a volume of **464ml. Z = 17 * 1047.2cm^2 / 464 = 38.4**

If you can't find steel pot scrubbers, metal turnings from a lathe work very well. It is impossible for us know the actual value of **Z** for lathe turnings, because they vary, but it is probably a bit less than 38.4. Since lathe turnings are generally bulkier, you couldn't get as much in a given length of column, but the comparison figures below show that there is plenty of leeway before efficiency drops.

Metal pot scrubbers are available in steel, brass and copper. We've talked about steel ones, because these are the most common. However, some people believe that copper is superior, for the same reasons it is used in whiskey stills. This is a fertile area for experimentation!

Comparisons

Z (surface area of packing / volume of column occupied by packing)

Marbles	**3.6**
Solid cylinders	**7.2**
Raschig rings	**14.4**
Pot scrubbers	**38.4**

Need we say more?

Surge boiling

Everyone knows what boiling is, and we discussed it at some length in Chapter 2, where we stated that boiling occurs when the vapor pressure of a liquid becomes equal to the external pressure exerted on it, and that the boiling point temperature therefore depended on what this pressure is. Now, we have to admit that reality is a bit more complex than that, and that the real situation is that when the vapor pressure of a liquid reaches the point where it's equal to the surrounding pressure, a bubble **can** form. **Can** – not **will**.

Before a bubble can form, a vast number of molecules must be "persuaded" to simultaneously move apart from each other to create the void we call "a bubble". Once the bubble is formed, evaporation into it is simple and the bubble can grow quite easily.

That first step, the formation of a bubble, is statistically an incredibly rare event! Without some external trigger, it's quite possible – indeed, common – for the temperature of a liquid to rise above its "normal" boiling point. The trigger that causes a bubble to form is called "nucleation", and may be one of many things. It may be a local disturbance caused by a crystal of salt or sugar dissolving, or dissolved air coming out of solution. It may be a tiny, localized hot-spot on a heater element, or even be a sharp edge on the surface of the container.

Pure water, which has been previously boiled to expel all dissolved air, is particularly prone to superheating if contained in something with a very smooth surface, like a glass or a glazed ceramic mug. Many people have been seriously burned when water in such a container was superheated in a microwave oven. The temperature of the undisturbed water can rise to alarming heights and, when disturbed in any way (even by a spoon tapping the side of the container) the superheated water can literally explode!

Fig. 8-11

Dr. Louis A. Bloomfield, Professor of Physics at The University of Virginia, has done remarkable experiments along these lines and published pictures and movies of the results on the website "howthingswork.virginia.edu" (select "microwave ovens" from the menu).

Surge boiling is a mild version of this explosive activity, and is usually encountered when heating a large volume of liquid with a thermostatically controlled heater. The initial boiling expels dissolved gases, removing a major nucleation source. When the thermostat turns the element off, the liquid cools and all bubbles collapse. During the next cycle of heating the bubbles that initially form appear only after the local temperature adjacent to the heating element has risen several degrees above the "normal" boiling point of the liquid. Since the element is usually placed at the bottom of the boiler, they are forming at higher than atmospheric pressure due to the depth of the fluid. These bubbles then trigger an avalanche of bubbles, which rapidly expand as they rise. The net result is a sudden surge of boiling each and every time the thermostat cycles, and large fluctuations in the rate at which vapor is formed. The end result can be like sitting your column on top of Old Faithful (which, in fact, operates in exactly the same way!)

This is why we advocate using a heat spreader with a hotplate, or an electronic (but compliant) power controller if using an immersed heater element. These methods allow you to maintain a steady simmer and a steady generation of vapor.

Supercooling and Column Stability

Hot vapor rises in a compound still to be condensed and returned to the column as reflux. How cold can this reflux be before operation of the column is upset?

Most people's initial reaction is to say, "Very little cooling can be tolerated – it may upset the equilibrium, and will certainly change the reflux ratio." This response is so common that it is almost unquestioned, and some people have gone to great lengths to design still heads to minimize reflux cooling. What can theory tell us about this?

First thoughts

Let's start by considering a column under 100% reflux, and assume for the moment that the condenser is "perfect" – any vapor touching it is condensed and its latent heat whisked away, but it doesn't cool the resulting liquid below the dew point.

The mass of the reflux being returned to the column will be equal to the mass of the vapor arriving. The temperature of the reflux will be the same as the temperature of the vapor in the top of the column, so this liquid reflux will trickle down through the packing until it reaches an area where the temperature is higher, at which stage it starts to vaporize. The liquid reflux does **not** start to vaporize as soon as it hits the top of the packing. As we showed in Fig. 8-10, it may well have to travel down the column some distance before it meets the higher temperatures that will initiate re-vaporization.

The equilibrium of the column has not been affected in the slightest, and the reflux ratio remains at 100%.

Now, reduce the temperature of the condenser so that the reflux is cooled by T degrees. Maintain 100% reflux.

When the cooled reflux re-enters the column, it will be surrounded by hotter vapor and begin to be warmed up, especially when it hits the top of the packing. Some of the rising vapor to condense, releasing latent heat which warms the cool reflux. While the cool reflux is being warmed, more liquid reflux is being formed by condensation and the reflux ratio rises **above** 100%. The quantity of this additional reflux is small, because the latent heat released by condensing vapor is much greater than the heat needed to warm the cool liquid reflux.

Let's say that the heat released by condensing X gm of vapor is sufficient to warm Y gm of cool liquid reflux back up to the ambient temperature in the top of the column.
So X times the LHV of the liquid equals Y times the specific heat S of the liquid times the temperature rise T degrees.
X * LHV = Y * S * T
So the ratio **Y:X = LHV:(S * T)**
Since the LHV is much Larger than S, Y is much larger than X.

Let's do the calculation for 95% ethanol, which has a latent heat of vaporization of 220.8 cal/gm and a specific heat of 0.601 cal/gm. This means that the ratio **Y:X** = 220.8 / 0.601 x T = **367 / T**. If we suppose the temperature drop is 10ºC (18ºF), then for every 37 gm of cool liquid reflux, just one gram of additional reflux will be formed by condensing vapor, and the reflux ratio will rise less than 3%. Not much of an impact so far!

Now consider how much vapor is left to carry on up to the condenser. It will be (Y-X) gm as X gm has been condensed. Let's call this y gm, so **y = Y-X**
This condenses and is cooled by T degrees by the condenser, and returns to the column to be warmed up.
Hot vapor condenses in this process, but now it will be only x gm, where **y:x** = **367 / T**
So **Y:X = y:x**
So not only will the amount of vapor reaching the condenser be less this time, but the amount of vapor condensed by cool returning reflux will also be less in the same proportion. However, as both **y** and **x** are smaller than **Y** and **X** respectively, **(y-x)** will be smaller than **(Y-X)**, and the reflux ratio will be closer to its original value of 100%

You can perform the calculation as many times as you wish, and add the results together to calculate the net total reflux, but a simpler way of looking at it is to imagine that the condenser has expanded into the topmost part of the packing. Once you consider the top bit of packing as the bottom of the condenser, you again have a "perfect" condenser like you started the analysis with.

The top part of the packing **automatically compensates** for any additional cooling, leaving the overall reflux ratio and the equilibrium of the column unaffected. This buffer zone is quite small, because the amount of extra condensation required to heat the cool reflux is so small.

Second thoughts

Not convinced? Let's do the multiple iterative calculations we mentioned before..

1) Start with N gm/sec vapor at 80ºC reaching condenser, and operate under full reflux, taking no product.
Now, increase condenser efficiency and let it cool the distillate by TºC.

This cool distillate is warmed up to 80ºC when it hits the buffer zone at the top of the packing, causing 0.601NT / 220.8 gm/sec of the rising vapor to be condensed.

We can simplify that by noting that for a given drop in temperature T, the relationship **0.601T / 220.8** is a constant. We'll call that constant 'd'.

So Nd gm/sec is condensed in the buffer. This joins the N gm/sec coming down as reflux, making the total reflux
(N + Nd), and the reflux ratio R = (N + Nd) / N

2) The vapor now reaching condenser is (N - Nd) gm/sec and this is condensed and cooled TºC. The rising vapor now condensed in buffer is now (N - Nd)d, and the total reflux is now (N - Nd) + (N - Nd)d = N - Nd + Nd - Nd^2 = N - Nd^2, and the reflux ratio R = (N - Nd^2) / N.

3) Now the vapor reaching the condenser is (N - (N - Nd)d = N - Nd + Nd^2 gm/sec and this is condensed and cooled T^o C. The rising vapor now condensed in buffer zone is (N - Nd + Nd^2)d, the reflux is now (N - Nd + Nd^2) + (Nd - Nd^2 + Nd^3) = N + Nd^3, and the reflux ratio R = (N + Nd^3) / N

4) This time the vapor reaching the condenser is N - (Nd - Nd^2 + Nd^3) gm/sec, and this is condensed and cooled T^o C. The amount of rising vapor now condensed in the buffer zone is (N - Nd + Nd^2 - Nd^3)d, and the reflux is now (N - Nd + Nd^2 - Nd^3) + (Nd - Nd^2 + Nd^3 - Nd^4) = N -Nd^4, making the reflux ratio R = (N - Nd^4) / N.

etc etc etc

Summary:
Reflux ratio is
1. (N + Nd) / N
2. (N - Nd^2) / N
3. (N + Nd^3) / N
4. (N - Nd^4) / N
.....
n. (N - $(-1)^n Nd^n$) / N

Since d < 1, this is a converging series with limit N÷N = 1
If T = 10 then d = 10 x 0.601 / 220.8 = 0.03, and the series converges very rapidly.
If T = 50 then d = 50 x 0.601 / 220.8 = 0.14. The series still converges rapidly, but takes a bit longer to settle.
The reflux oscillates around a mean of N. This oscillation is rapidly damped, and therefore inherently stable.

Now take off product between condenser and buffer at rate "r" gm/sec

1)
Vapor initially reaching condenser is N gm/sec
Cooled distillate reaching buffer is (N - r) gm/sec
Rising vapor condensed in buffer is (N - r)d gm/sec
Reflux ratio R = (N + Nd - rd - r) / N
= [N - r]÷N + [(N - r)d] / N

2)
Vapor now reaching condenser is N - (Nd - rd) = N - Nd + rd
Cooled distillate reaching buffer is N - Nd + rd - r
Rising vapor condensed in buffer is (N - Nd + rd - r)d
Reflux ratio R = [(N - Nd + rd - r) + (Nd - Nd^2 + rd^2 - rd)] / N
= [N - r - Nd^2 + rd^2] / N
= [N - r]÷N - [(N - r)d^2] / N

3)
Vapor now reaching condenser is N - (N - Nd + rd - r)d = N - Nd + Nd^2 - rd^2 + rd
Cooled distillate reaching buffer is N - Nd + Nd^2 - rd^2 + rd - r
Amount condensed by this is (N - Nd + Nd^2 - rd^2 + rd - r)d
Reflux ratio R = [N - Nd + Nd^2 - rd^2 + rd - r] + [Nd - Nd^2 + Nd^3 - rd^3 + rd^2 - rd]
= [N - r +Nd^3 - rd^3] / N
= [N - r]÷N + [(N - r)d^3] / N

etc etc etc

Summary:
Reflux ratio is
1. [N - r]÷N + [(N - r)d] / N
2. [N - r]÷N - [(N - r)d^2] / N
3. [N - r]÷N + [(N - r)d^3] / N
.....
n. [N - r]÷N - $(-1)^n$ [(N - r)d^n] / N

Once again, since d < 1, then this is a converging series with limit [N - r] / N
The reflux oscillates around a mean of [N - r], and this is a rapidly damped oscillation and stable as before.

This shows that the system exhibits damped oscillation should a sudden change in cooling be imposed, but rapidly settles down to the "default" reflux ratio that would exist should no cooling occur. It also shows that this occurs no matter how severe the initial cooling may be, large oscillations taking only a little more time to damp out.

They were wrong!

A compound column is inherently stable in respect of the temperature of the reflux returned by the head condenser, so no special precautions need be taken to control the flow of coolant in that condenser, other than keeping it steady at a rate that ensures complete condensation of all vapor reaching it. Many people have spent needless hours worrying and struggling to design systems that avoid cooling the condensate, and all the time they were pursuing the wrong goal. Some time spent up front studying theory can save many hours of fruitless toil.

(If you're using Cooling or Vapor Management (Chapter 4) to control reflux, then of course you **don't** want complete condensation, but the key point is that the stability of the system ensures that whatever reflux ratio you do impose will remain constant.

A Final word

There now! In this, the last chapter, we have discussed subjects ranging from basic physics and chemistry to the design and operation of fractionating stills, stopping along the way to examine the reasons why they are built and operated the way they are. We hope that this journey has been interesting and illuminating, and that it has given you at least a few ideas that you will follow up as you enjoy the activities involved in distilling. We sincerely believe that the more deeply you understand the processes you use, the more enjoyable and controllable they are, and the better the products you will make.

Remember the words we paraphrased from Isaak Walton in the Foreword of this book?

"Doubt not, therefore, sir, but that distilling is an art,
and an art worth your learning."

Enjoy your Art, as we hope you have enjoyed this book.

APPENDIX 1

WEIGHTS AND MEASURES

There are many different systems of weights and measures in use around the world, and it's often difficult to translate from one to another. Fortunately, the metric system is in use almost everywhere on Earth, and the American and Imperial systems are widely familiar. We shall use the SI metric system as our baseline, and provide conversions to the American and Imperial measurements, where it would be useful. The American and Imperial systems share the same weight and distance measurements, differing primarily in liquid measures.

The history of why the Imperial gallon differs from the US gallon is interesting. The Queen Anne (1707) wine gallon measured 231 cubic inches, yet the ale gallon most commonly in use (there were several) measured 282 cubic inches. When the Americans achieved independence in 1776 they had to adopt a system of weights and measures and chose to use the Queen Anne gallon for liquid measures. In contrast, the British chose to follow the ale gallon route when, in 1824, they defined the Imperial gallon as being that which contains 10 pounds avoirdupois of distilled water at 62 deg. Fahrenheit and barometer of 30 inches – a volume of 277.42 cubic inches. To further complicate matters, the Americans chose to have 16 fluid ounces (floz) in each of their pints, but the British chose to have 20. Oddly enough, this made the two fluid ounces almost identical!

We end up with the following conversion table:

Volume	**US**	**Metric**	**Imperial**
	1 US gallon	3.785 liter	0.833 Imp gallon
	1 US quart	946.35 ml	0.883 Imp quart
	1 US pint	473.18 ml	0.833 Imp pint
	1 US floz	29.57 ml	1.041 Imp floz
	US	**Metric**	**Imperial**
	33.81 US floz	1 liter	35.2 Imp floz
	0.26 US gallon	1 liter	0.22 Imp gallon
	1.06 US quart	1 liter	0.88 Imp quart
	2.11 US pint	1 liter	1.76 Imp pint
	US	**Metric**	**Imperial**
	1.201 US gallon	4.546 liter	1 Imp gallon
	1.201 US quart	1.137 liter	1 Imp quart
	1.201 US Pint	568.26 ml	1 Imp pint
	0.96 US floz	28.41 ml	1 Imp floz

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Mass

1 ounce	=	28.35 gram
1 pound	=	453.6 gram
1 gram	=	0.035 ounce
1 kilogram	=	2.2 pound

Force

It is important to distinguish between **Mass** and **Force**.
Mass is a physical property of material, a basic measure of how much "stuff" the material contains.

If you apply a **Force** to an object, it will accelerate. The more force, the greater the acceleration. The more mass the object has, the more force required to get the same acceleration. The amount of force required to accelerate 1 gram to a speed of 1 centimeter per second in one second is called a **dyne**, and the amount of force required to accelerate 1 kg to a speed of 1 meter per second in one second is called a **Newton**.

The **Weight** of an object is the force exerted on its mass by gravity. On the surface of the Earth, a 1kg mass sitting on a table exerts 1 kg-force downward. This force is what we measure as weight. Gravity accelerates objects to a speed of 9.8 meters per second in one second, so 1 kg-force is equal to 9.8 Newtons.

When objects are removed from gravity (eg on the space shuttle in orbit), they are weightless, but still have their mass. This is why astronauts handle large weightless objects so carefully – they take a lot of force to start moving, and just as much force to stop again.

Pressure

Pressure is force per unit area. Measured in pounds per square inch, etc.

1 atmosphere	=	14.7 lb/in^2
	=	760 mm of mercury (also called Torr)
	=	101.325 kilopascals
	=	1013.25 millibar

Length

1 inch	=	2.54 centimeter
1 foot	=	30.48 centimeter
1 centimeter	=	0.39 inch
1 meter	=	39.37 inch

Temperature

Two things are important in temperature calculations: the actual temperature, and the difference or change in temperature.

Temperature Difference	**Actual Temperature**
change (ºC) = 5/9 change (ºF)	(^oC x 9/5)+32 = ^oF
50ºF change = 28ºC change	(^oF-32)x5/9 = ^oC
change (ºF) = 9/5 change (ºC)	0 ^oC = 32 ^oF
15ºC change = 27ºF change	100 ^oC = 212 ^oF

Electrical relationships

	voltage	=	current x resistance	[volts = amps x ohms **V = IR**]
	power	=	voltage x current	[watts = volts x amps **W = VI**]
thus:	power	**W =**	**$VI = V^2/R = I^2R$**	

Work, Energy and Power

Work is the mechanical expression of energy. In scientific terms, it is the exertion of force over a distance. For example, if you raise a 1 kg mass 1 meter, you have done 9.8 **Newton-meters**, or **Joules** of work. Work and energy are measured in the same units.

Energy is the capacity to do **Work**, and it may be exist in many different forms. Commonly encountered forms of energy are **potential** (a heavy weight on a stand contains potential energy), **kinetic** (yank the stand out from under the weight, and its movement is kinetic energy), and **heat** (when it crashes to the floor, the damage it does ultimately turns to heat).

Power is the rate at which work is done. One Joule of energy applied for 1 second is a Watt.

Here are the main conversion factors that relate to heat and electrical energy.

The basic unit of energy and work is the **Joule.**
If one Joule of energy is expended every second, the power level is 1 **Watt**,

- **1 Watt = 1 Joule/sec = 60 Joules/min = 3,600 Joules/hour**

The **calorie** is a common measure of heat energy equivalent to **4.1852 Joule**.

- **1 Watt = 3,600 ÷ 4.1852 = 860 calories/hour**

Physical relationships

Standard Temperature and Pressure (STP) = 760 Torr and 0ºC.
1 gram mole = The amount of a substance whose mass in grams is equals the mass of one molecule in atomic mass units (approximately 6.022 x 10^23 molecules).
Degrees Kelvin (absolute temperature) $^{o}K = {}^{o}C + 273.16$
Universal Gas Constant, R = 8.314 kJ/mol.^{o}K
Gas equation: pressure x volume = R x absolute temperature [PV = RT]
1 gram mole (mol) of vapor occupies 22.4 liter at STP

Water:	Boiling point (SAP)	= 100^{o}C
	Density	= 1 gm/ml at 16^{o}C
	Surface tension	= 54.9 dyne/cm^2 at 40^{o}C
	Specific heat	= 1 cal/gm ^{o}C
	Latent heat	= 540 cal/gm = 40.639 kJ/mol
	Molecular mass	= 18.016 atomic units
Ethanol:	Boiling point (SAP)	= 78.5^{o}C
	Density	= 0.791 gm/ml at 16^{o}C
	Surface tension	= 21.38 dyne/cm^2 at 40^{o}C
	Specific heat	= 0.58 cal/gm.^{o}C
	Latent heat	= 204 cal/gm = 39.33 kJ/mol
	Molecular mass	= 46.068 atomic units

pH

Water molecules are constantly splitting apart ("dissociating") into a hydrogen ion (H^+) and a hydroxyl (OH^-) ion, and just as rapidly, these ions recombine to form water. Acidity is produced by hydrogen ions and alkalinity is produced by hydroxyl ions. In pure water, the concentration of hydrogen ions and hydroxyl ions is equal, and the solution is neutral - neither acidic nor alkaline.

- In pure water, the equilibrium concentration of H^+ and OH^- is 0.0000001 (10^{-7}) gram moles per liter. Gram moles per liter of H^+ is written $[H^+]$.
- Some dissolved substances affect the relative concentration of H^+ and OH^-, making the solution acidic or basic.
- Since H^+ and OH^- will recombine whenever possible, when H^+ goes up, OH- must go down, and vice versa.
- H+ and OH^- concentrations can vary through a **huge** range, from 1 gm mole/liter to 0.00000000000001 (10^{-14}) gm mole/liter.
- To deal with ranges of numbers this large, scientists usually use logarithms, or powers of 10. In typical laboratory solutions, $[H^+]$ may vary from about 0.01 gm mole/liter (10^{-2}) to 0.0000000000001 gm mole/liter (10^{-13}).
- It is easier to write **the pH range is from 2 to 13**

So, the **pH** of a solution is the negative logarithm of the hydrogen ion concentration, **$pH = -\log[H^+]$**

If the hydrogen ion concentration is increased, the pH drops, and the solution becomes more acidic.

If the hydrogen ion concentration is decreased (increasing $[OH^-]$) the pH rises, and the solution becomes less acidic; in other words, more basic (alkaline). Since the system is based upon powers of 10, a pH of 4 is ten times more acidic than a pH of 5, and a pH of 10 is ten times more basic than a pH of 9.

APPENDIX 2

CARBOHYDRATES FOR FERMENTATION

Carbohydrates and their chemistry can be an extremely complicated subject. In this appendix, we're going to give you just a taste of that complexity, and then simplify back down the few simple points that apply directly to fermentation and distillation. Knowing about the complexity and the vast numbers of different kinds of sugars can help appreciate how valuable the traditional fermentable sources truly are.

Carbohydrates are materials made up exclusively of carbon, hydrogen and oxygen, in a ratio that is very close to one atom of oxygen and two of hydrogen for every carbon atom. The general carbohydrate formula is $C(H_2O)n$. Chains of three or more carbon molecules are called **sugars**, and are given names that end in the letters **-ose**. Sugars with six carbons (the most common size) are called hexoses, and five-carbon sugars are pentoses.

Many different sugars have exactly the same chemical formula – they are isomers of one another, differing by the arrangement of the atoms on the chain. Here are the chemical structures, in chain form, of several different hexoses, all with the same formula, and the same molecular weight – 180.16. Of these, only glucose and fructose are readily fermentable.

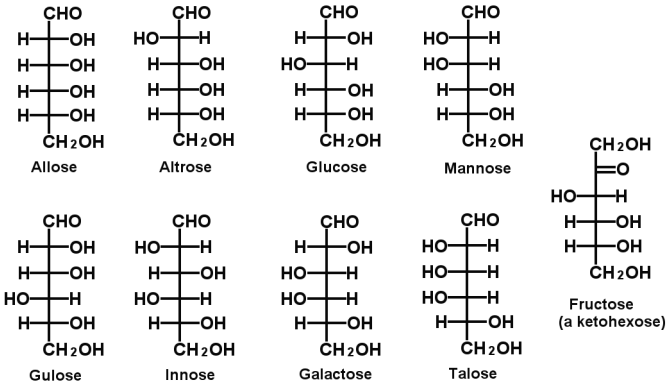
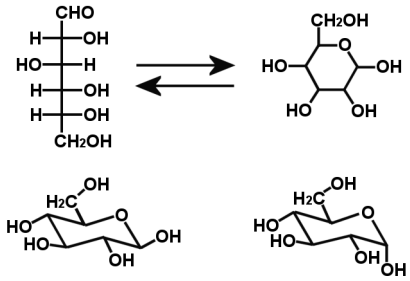


Fig. A2-1 AldoHexoses

To make things more complicated, each of these hexoses can form rings due to the interaction of oxygen molecules, and they can form several different kinds of rings. Here is an example of how this happens in glucose



The rings form and fall apart constantly, so all the different forms exist in a state of dynamic equilibrium. Here are diagrams of the various forms of glucose and fructose.

Fig. A2-2 Glucyclation

And to get one step more complicated, most of the ring forms can exist in different structural versions.

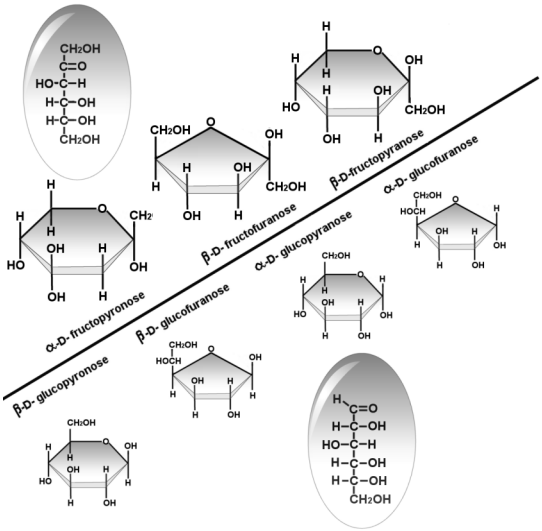
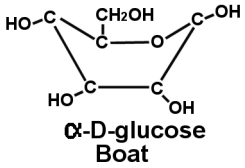


Fig. A2-3 Fructomers and glucomers

Here are the two forms of (alpha)-D-glucose.



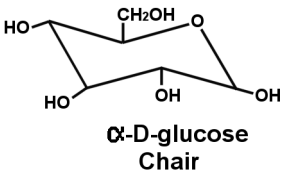


Fig. A2-4 Glucboat and Gluchair)

Since enzymes interact with molecules on the basis of their shape, any particular enzyme usually only interacts with one of the many forms a sugar molecule can assume.

Fortunately, all we really need to remember is that fructose and glucose are the fermentable simple sugars, and that yeast can readily move these molecules into the cells for processing into ethanol and carbon dioxide.

Sugar molecules can join together into chains in a variety of forms to form **polysaccharides**. Groups of two sugar molecules are called **disaccharides**, three are **trisaccharides**, and so forth. Simple polysaccharides with 10 to 50 sugar molecules are collectively called **dextrins**. Polysaccharide molecules can be straight or branched, and can become very complex.

The most common disaccharide is **sucrose** (common table sugar), which is composed of one glucose and one fructose molecule. In the process of joining together the two hexoses, one molecule of water is given up, so the molecular weight of sucrose is 342.3 – 5.2% lower than the combined molecular weight of fructose and glucose. Another common disaccharide is maltose, formed from two glucose molecules, and a common trisaccharide is maltotriose is a glucose trisaccharide. Here are pictures of these three fermentable sugars.

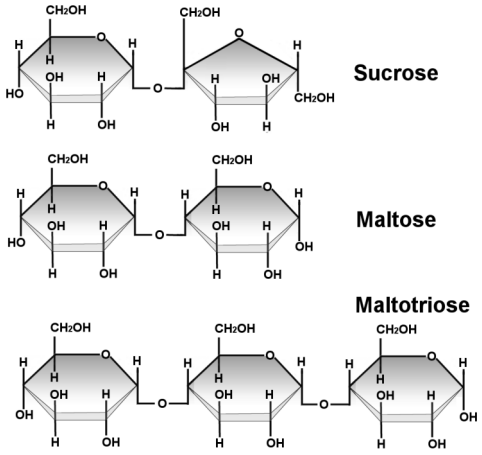


Fig. A2-5

Sucrose cannot be transported across cell walls, but some types of yeast and bacteria release enzymes into their environment that **hydrolyze** sucrose, adding back the missing water molecule, and producing one molecule of glucose and one of fructose. These are then taken into the cell and processed.

Many kinds of polysaccharides cannot be enzymatically broken down, and thus cannot be fermented. The dextrins produced in a beer-making wort are not fermentable, and give the beer its body. A few special types of carbohydrates are very widely made by plants. Two of the most familiar are starch and cellulose, both of which are made up of chains of glucose molecules. Starch and cellulose differ in how they are linked together. Starch is built in exactly the same way as maltose and amylose, with every glucose molecule joined to the next on their "lower" side (this is called an "alpha" linkage). In cellulose, the molecules are joined together from the lower side of one to the upper side of the next, called a "beta" linkage. Although the diagram shows all the sugar molecules "right side up", in the beta linkage, every other one is actually "upside down". Here are diagrams of starch and cellulose.

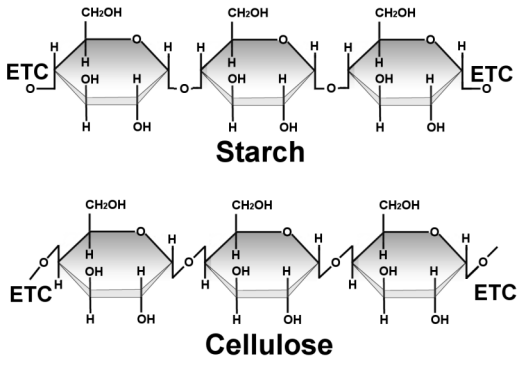


Fig. A2-6

The alpha linkage of starch makes it fairly easy for enzymes to break down into glucose, which is why plants use starch as a "storage material" for food energy. When they need stored energy, they release enzymes called **amylases** which break down the starch. We use these same enzymes to prepare starchy material for fermentation.

Cellulose's beta linkage, on the other hand is much harder to break down, and it is used by plants as a primary structural material. There are a few organisms that make enzymes capable of hydrolyzing cellulose, but the process is very slow and expensive compared to starch.

Pectin is another complex polysaccharide that should be of interest to beverage makers. Pectin is mainly composed of **methyl esters of galactose**, joined by a beta linkage. It is a primary constituent of plant cell walls, and is especially abundant in fruit. The methyl esters are the "$COOCH_3$" groups in the picture below. This represents (O=C-**O-CH_3**). The non-methylated sugars have "COOH" (O=C-OH) in the same location. These two forms are mixed together in no particular order, and in amounts that vary.

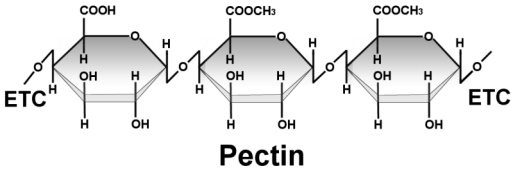


Fig. A2-7

When pectin is broken down by micro-organisms, some of the the methylated residue group combines with water (hydrolysis) and changes to the non-methylated group, releasing methanol, which is poisonous

O=C-O-**CH_3** + H-**OH** → O=C-OH + **CH_3-OH**

Any distillation of fruit-based ferments will produce foreshots containing methanol, which is why they should be discarded. Wort derived from grain contains very little pectin, because it remains in the grains. Whole grain mashes will produce some methanol. Purified sugar does not contain any pectin, and **sugar-based washes do not produce any methanol**. However, the foreshots will contain other unpleasant substances, such as ethyl acetate, and should also be discarded.

APPENDIX 3

HYDROMETER TABLE

A. **Grams sucrose per liter solution**
B. **Specific Gravity at 60º F(15.6º C)**
C. **Percentage sucrose by weight (Degrees Plato, Brix or Ballings)**
D. **Degrees Oeschle**

A	B	C	D
10	1.0039	1.0	4
20	1.0077	2.0	8
30	1.0116	3.0	12
40	1.0155	3.9	15
50	1.0193	4.9	19
60	1.0232	5.9	23
70	1.0271	6.8	27
80	1.0309	7.8	31
90	1.0348	8.7	35
100	1.0387	9.6	39
110	1.0425	10.6	43
120	1.0464	11.5	46
130	1.0502	12.4	50
140	1.0541	13.3	54
150	1.0580	14.2	58
160	1.0618	15.1	62
170	1.0657	16.0	66
180	1.0696	16.9	70
190	1.0734	17.8	73
200	1.0773	18.6	77
210	1.0812	19.5	81
220	1.0850	20.4	85
230	1.0889	21.2	89
240	1.0928	22.1	93
250	1.0966	22.9	97

A	B	C	D
260	1.1005	23.8	100
270	1.1044	24.6	104
280	1.1082	25.5	108
290	1.1121	26.3	112
300	1.1160	27.1	116
310	1.1198	27.9	120
320	1.1237	28.7	124
330	1.1275	29.5	128
340	1.1314	30.4	131
350	1.1353	31.2	135
360	1.1391	31.9	139
370	1.1430	32.7	143
380	1.1469	33.5	147
390	1.1507	34.3	151
400	1.1546	35.1	155
410	1.1585	35.9	158
420	1.1623	36.6	162
430	1.1662	37.4	166
440	1.1701	38.2	170
450	1.1739	38.9	174
460	1.1778	39.7	178
470	1.1817	40.4	182
480	1.1855	41.2	186
490	1.1894	41.9	189
500	1.1933	42.6	193

APPENDIX 4

ACTIVATED CARBON

Removing unwanted substances from liquids and gasses by filtering them through a solid material is an ancient technique. The process of adsorption was described as early as 1550 BC in an ancient Egyptian papyrus and later by Hippocrates and Pliny the Elder. It was mainly used for medicinal purposes.

At the beginning of the 19th century, the ability of bone char to take the color out of liquids was discovered by the sugar industry in England. At the beginning of the 20th century, methods of **activating** carbon with chemicals or by use of steam were discovered. During the First World War, steam activated coconut char was developed in the United States for gas masks. Today, many different materials – not only carbon – can be processed to develop selective adsorption of a wide variety of substances, and these techniques are used extensively in many industrial processes and the manufacture of foods, beverages, and medicines.

Adsorption

Many people confuse absorption and adsorption because the words sound similar, but they are very different processes. When a material is **ab**sorbed, it is held within a porous material by capillary action. A sponge absorbs water, and releases most of it when squeezed. A piece of chalk or a brick will also absorb water, but clearly demonstrate that absorbed materials aren't always easily removable.

Adsorption, on the other hand, is a process involving intermolecular and electrostatic forces.

All atoms and molecules are characterized by the way in which their electronic charge is distributed. There are a variety of ways for charge to distribute, and these help define the chemical and physical properties of the molecule. One of these effects, **polarity**, helps define the solvent properties of a compound. Another, **resonance**, is frequently associated with colored organic molecules. Each of these effects also creates unique possibilities for electrostatic attraction between molecules, and these attractive forces can pull molecules together from distances several times the size of the molecule.

At very close range, another attractive force exists between all atoms and molecules. These are called the **van der Waals** forces, named after the physicist who discovered them. Van der Waals forces are quite powerful once the molecules have been brought close enough together for them to act.

Carbon atoms in charcoal can join together in a wide variety of forms with widely different electron distributions, meaning that it will have areas on its surface that are attractive to almost every kind of molecule. Once electrostatic attraction has pulled a molecule close to the surface of the carbon, van der Waals forces take over and hold it in place tightly. Charcoal also has millions of tiny pores in its structure, which create a very large surface area for adsorption to occur.

Treatments of the carbon or charcoal can alter the distribution of pore sizes and of electrical effects, "tailoring" the carbon to primarily attract and hold certain types of compounds.

Activation

Activation is the process of processing a material that is intrinsically adsorbent, materials such as zeolite or carbon, to enhance its adsorbent properties. Heat and steam treatment activates carbon by helping to create large numbers of small pores of a selected size, and chemical treatments create the proper electrostatic environment to attract and hold onto specific molecules. The most effective activated carbons receive both steam and chemical treatment, and the millions of pores together give in an enormous internal surface area enabling large quantities of selected molecules to be held. Both the electrostatic and the van der Waals forces are weak enough that the adsorption process is reversible by heating the carbon. This makes the process practical and economical, because the carbon can be re-used many times.

We tend to think of activated carbon as being primarily useful for removing the molecules that contribute to unpleasant flavors in beverages, or for removing noxious gases when used in respirators. However, highly colored organic compounds tend to have strong electrical charges and are readily adsorbed by activated carbon, making it useful for removing both unpleasant flavors and unwanted colors. One of the first uses of activated carbon was to decolorize the raw product of the sugar industry and make it look more palatable.

Most activated carbon is prepared from plant or animal material, treated to remove everything but the carbon in its structure. Heating these materials in the absence of air converts them to charcoal, which retains much of the porous structure of the biological cells. Unfortunately, it also retains all the salts and many other non-volatile compounds that were present in the original material. The activating process removes many of these compounds, creating a purer and more effective carbon for adsorption, but also leaves behind other salts and residues.

Activated charcoal is prepared in three main forms:

- As granules – irregular shaped particles with sizes ranging from 0.2 to 5mm. This type is used for treating both liquids and gases.
- As powder – pulverized carbon with a size predominantly less than 0.18mm (US Mesh 80). This type is mainly used for treating liquids and for flue gas treatment.
- As pellets, or "prills" – extruded cylindrical shapes with diameters from 0.8 to 5mm. This type is mainly used for gases (but can be used for liquids too) because they offer least resistance to gas flow, have high mechanical strength, and low dust content.

Preparation for use

The salts and chemical residues that may be present in activated carbon can spoil the flavor of a beverage treated with them. Always wash new carbon **thoroughly** before using it. The only effective way of washing activated carbon is to boil it for at least 5 minutes in fresh water, then rinse it in several changes of water, then repeat the boiling and rinsing two more times. This treatment will assure that you have pure activated carbon that will not leave any tastes or residues in your ethanol.

How to use activated carbon

Activated carbon may be used as a filter bed, passing liquid through it in a slow, steady stream. This is very common industrially, where materials are passed through several beds of activated adsorbents as part of a continuous flow process. However, the longer a liquid is in contact with an adsorbent, the greater the degree of purification. If you are treating alcohol with a filter bed, you should dilute it to approximately 40% before the treatment. This is because the addition of water makes the congeners less soluble, and easier for the carbon to adsorb.

If you have the time and space, it is far more effective to simply place your alcohol and the activated carbon together in a large container and let it sit for a couple of weeks. This method works very well with alcohol of any strength. We have regularly used this method with 96% ethanol with very good results.

With either method, you will need to filter the alcohol carefully after carbon treatment to remove tiny carbon dust particles. A few cotton balls in the neck of a large funnel does a fine job.

Cleaning used carbon

We mentioned earlier that the adsorption process could be reversed with heat. You can use this de-sorption process to re-activate used carbon. A significant amount of heat is required; re-boiling the carbon will not do the job. Industrially, activated carbon is cleaned with steam, or by baking in an oxygen-free oven at temperatures as high as 800ºC (1472º F).

One of the best ways to re-activate carbon is with steam. You can pack the carbon into a metal tube and blow hot steam through it under pressure, which is very efficient because the flow of steam carries the volatile contaminants away. However, you need at least one atmosphere of over-pressure and temperatures of 120ºC (250ºF), which makes this a dangerous process to try at home.

Alternatively, you can put the carbon into a pressure cooker with a little water. Boiling under pressure, and therefore temperatures higher than 100°C, for half an hour will ensure that the deepest pores of the carbon are washed to remove most of the volatiles, and what few remain can be baked out further by an hour or so in a very hot oven (260ºC, 500ºF). The water remaining in the carbon when you take it from the pressure cooker will help carry out the majority of the volatiles when it's first put in the hot oven, as the presence of water will depress the combined boiling point. After that, the high temperature does the rest.

Alternatively, you can leave the pressure cooker alone and simply spread the carbon out in a thin layer on a baking sheet and heat it in the oven. As noted above, it helps if you first make sure the carbon has been thoroughly washed in water and is still wet when first put in the oven. This is usually good enough for most purposes. However, do be sure that your oven is reasonably clean, or you may end up with carbon that smells like the last Sunday roast!

The strong smell coming from the pressure cooker and/or the oven is your proof that the process works, and an indication of what you will **not** be drinking!

These processes all work very well, removing the majority of contaminants and allowing you to re-activate your carbon as many times as you like. Over time, the carbon granules will physically break up and become dust, but if you don't mind the additional time it takes to filter off these small particles, just remember what was said above about carbon powder – pulverized carbon is used to clean liquids.

Before using the re-activated carbon, **boil it** in fresh water to degas it and make sure that the pores are all filled with liquid. Just wetting the carbon is not enough. Gas retention will greatly diminish the effectiveness of the carbon as the molecules in the liquid will be unable to diffuse into the deepest pores.

APPENDIX 5

CONSTANT LOAD HEATER CONTROLLER

This appendix is written for those who already have practical experience in electronics, so we don't spend time explaining what all the components are and how they work. If you don't have such experience, then this appendix may perhaps stimulate interest in yet another useful and absorbing hobby. Only one small part of the circuit discussed here has any direct connection with power supply voltages, so if you get a friend who does have experience in electrical circuits to first check what you have done before connecting this part to the main power supply, then there is no reason at all why you shouldn't have a go at making the controller yourself.

Operation

This controller provides fully variable heater element control by means of burst fire control of two triacs, one feeding power to the heater element under control, and the other to a dummy load with the same wattage as the heater element. Another heater element of the same value as the boiler heater element is the obvious choice, heating a quantity of water in a separate container. As was noted in Chapter 4, the water in the separate container will heat up very slowly, as it's only absorbing heat energy not required for the distillation process. To save you looking that up again, it was worked out that it would take over 3 hours to heat 50 liters of water in such a "dummy load" from 20°C to 60°C. The reason for wanting to do it this way will be explained later.

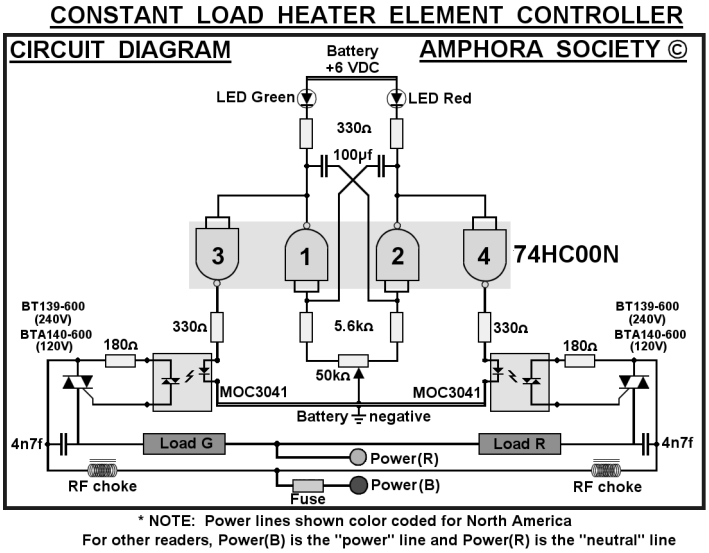
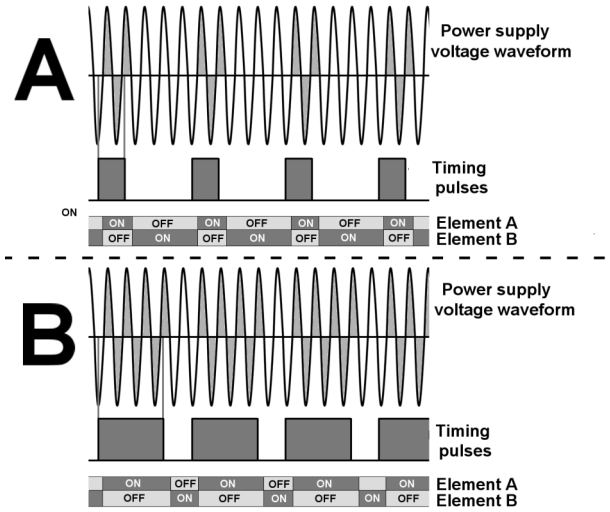


Figure A5-1 Schematic

The heart of the control circuit is the 74HC00N, a CMOS chip that contains four 2-input NAND gates. The inputs of each gate are shorted to make them operate as inverters. When the input goes high (6 volt) then the output goes low (zero volt), and vice versa. There are inverter chips available and they may be used with equal facility if you wish. The first two gates, 1 and 2, are cross-connected via an RC circuit to form an astable multivibrator. The duty cycle of this is around 2 seconds with the components shown, this being chosen as anything longer might lead to surge boiling. The mark-space ratio is controlled by means of the 50kohm variable resistor. The duty cycle time is not affected as this is connected as part of the resistor 'tail' for each side of the multivibrator. The switching cycle is visually displayed by the two monitor LEDs.

The two other gates, 3 and 4, are used to control the LEDs in the two MOC3041 optoisolator zero crossing triac triggers. Output is taken from these gates rather than from the nose terminals of gates 1 and 2 to ensure that triggering is unaffected by any strike resistance variations in the monitor LEDs. The small triac gate inside the MOC3041 is biased on whenever the LED is lit and so that triac conducts when the built-in zero-crossing detector determines that the power supply voltage is zero and going positive. This then triggers the power triac BT139-600 (240V power supplies) or BTA140-600 (for 120V power supplies) to switch on and supply power to the heating element. If you look carefully at Fig. A5-1, you will see that switch-on is initiated by the timing pulse going positive, but is triggering of the triac is delayed by the zero crossing detector in the MOC3041 until the power supply voltage crosses zero. Switch off is also delayed after the timing pulse falls to zero, the triac switching itself off when the power supply voltage falls to zero. All switching of the power supply is therefore done at zero voltage, and sudden voltage ramps that generate EMI are avoided.



Power is thus switched from one load to the other in time with the duty cycle and mark-space ratio of the multivibrator, and this varies the average power delivered to each heating element. Also, by ensuring that when one heater element is switched on the other is switched off, and vice versa, a constant load is presented to the main power supply.

Figure A5-2 Waveforms

New regulations

Constant loading is very important as frequent and regular switching of large power loads can cause power supply flicker. Indeed, CENELEC, the European Committee for Electro-technical Standardization, has published rules which limit the rate at which domestic heating apparatus may be switched on and off. This table (from CENELEC publication EN50.006) gives the minimum repetition period for a range of load powers and common power supply voltages:

Appliance	Repetition period, in seconds		
Power (W)	220V	240V	380V
600	0.2	0.2	-
800	0.8	0.3	0.1
1000	2.0	1.0	0.2
1200	4.6	2.0	0.2
1400	7.0	4.3	0.2
1600	10.0	6.3	0.3
1800	16.0	8.9	0.5
2000	24.0	13.0	0.9
2200	32.0	17.0	1.3
2400	40.0	24.0	1.9
2600	-	31.0	2.6
2800	-	-	3.6

By using a "dummy" load, we ensure that a constant loading is presented to the main power supply and the circuit complies with these rules. For American readers, do not imagine that these rules will not apply to you simply because the voltages from this European standard do not include 115/120V. You may deduce from the figures that the harmful effects of repetitive loading will be felt even more in low voltage power supplies, and if similar regulations have not yet been enacted in the USA, you may be assured that they are on their way!

Fig. A5-2 CENELEC Regulations

Zero crossing and EMI

As switch-on for each heating element occurs when the MOC3041 chip detects rising zero crossing of the main power supply, and switch-off of the power triac naturally occurs when the voltage falls to zero crossing at the end of a half cycle of that supply voltage, sudden voltage ramps are avoided. Sudden voltage ramps occur during every half cycle of the main power supply when triacs are phase controlled, as in small light dimmers, and this is a major source of electromagnetic interference, EMI. It is therefore totally irresponsible to use such "dimmer" circuits to control large power loads unless the controller is certified as being fully and effectively screened to eliminate EMI. Switching on and off at zero crossing, and then only twice every two seconds, greatly reduces the possibility of EMI. However, as no zero crossing detector circuit is perfect, and we want to suppress even tiny amounts of EMI that may occur, we've included an RF choke in our controller circuit to be on the safe side. In this manner, with EMI suppression and with constant loading on the power supply, we've ensured that this controller is as "clean" as it's possible to get with a home-made device.

The Circuit

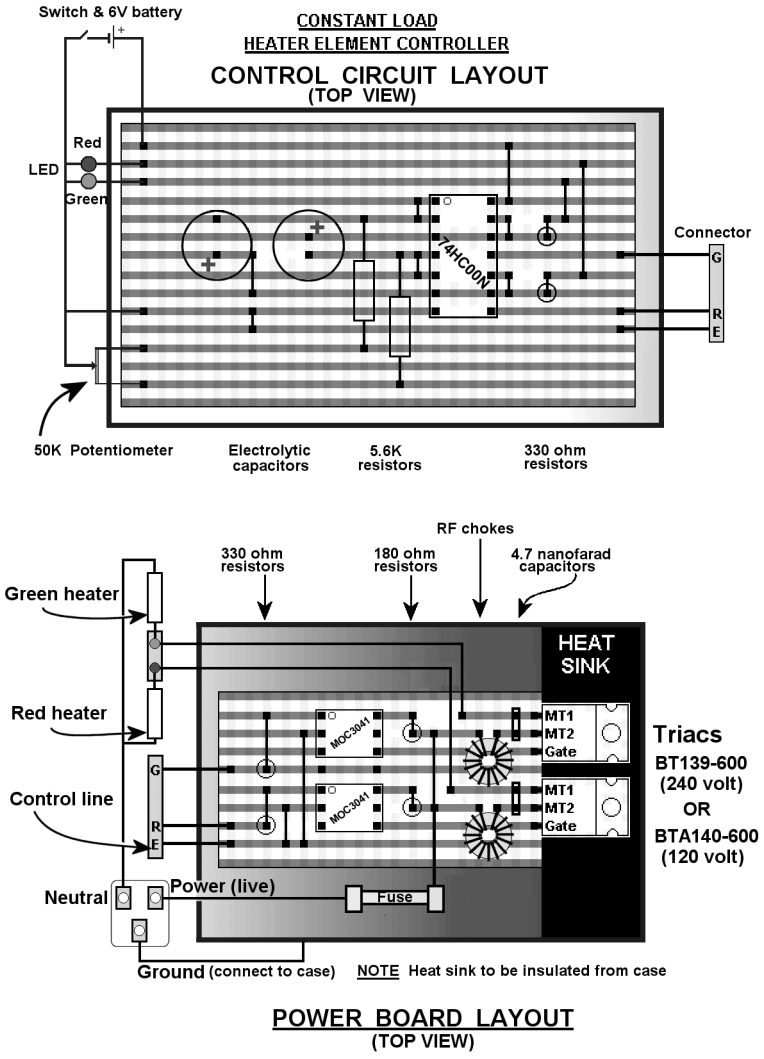


Figure A5-3 Circuit boards

We have designed the circuit around pre-drilled stripboard (Veroboard® type)as this requires no skill at making PC boards. If you know how to etch boards, then you may well end up with a neater arrangement. However, please do not fall into the trap of trying to cram everything into as small a space as possible. That is silly and counterproductive, and dangerous when dealing with the power board.

In the diagram, we have completely removed the lands between terminals where breaks are indicated as this makes it clearer to see, but just removing a small circle with a 4mm drill bit to break the land is all that needs to be done in practice.

All resistors are 1/4 watt carbon and both the 74HC00N and MOC3041 chips are mounted on 14 pin dil sockets.

Safety

The control circuit is isolated from all power supply voltages, not only by the optoisolator but also by distance. Connection to the power module is by means of around a couple of metres of 3-core wire. This carries only tiny currents at dry cell battery voltage, so the type of extension lead used with portable tape or CD players is ideal. However, please ensure that the power module is **very** well insulated and safe to handle with its covers on.

Triacs will typically emit around 1 watt of heat for every ampere of current being passed, so the power triacs must be mounted on a good heat sink and be well ventilated. Remember that the body of the triac is at power supply voltage, so the heat sink will be **live**!!! Insulating with mica strips is no assurance that this is not so! We strongly recommend mounting the power module in a plastic or wooden box with ventilation holes and fitted with proper power supply sockets.

The circuit is rated to handle up to 2 kW. Care should be taken to ensure that you select the correct power triacs for the power supply voltage you will be using. A BTA140-600 will handle currents up to this rating with both 115/120V and 240V power supplies, and a 20 amp fast-blow fuse should be used with it.
However, a BT139-600 should be used for 240V power supplies **only**, and with a 10 amp fast-blow fuse.

Extreme care needs to be exercised in connecting suitably rated wiring to the MT1 and MT2 terminals of the power triacs. Remember - these wires will be at mains voltages and carrying the full load current of the heater elements. The current at 115V will be more than twice the current at 240V, and at 2 kW these currents will be 17.4 amp and 8.4 amp RMS respectively. In other words, **peak** currents of 25 amp and 12 amp respectively! It doesn't take much resistance in a badly soldered joint to generate a lot of heat at these currents. Remember, the heat in watts generated will be the **square** of the current times the resistance! For this reason, "plug-in" connectors should not be used for the power triacs. They must be secured with good, sound soldered connections. All other "chips" may be plugged into dil sockets, which makes life easy, but you should spend some time and care ensuring that the power triacs are carefully soldered in place. You will therefore need to use a clip-on heat sink to avoid damaging the delicate electronics inside the power triacs when you solder them in place.

The RF choke

The RF choke is the only component you will have to make yourself, but a quick look at the diagram will show that this is very easy.

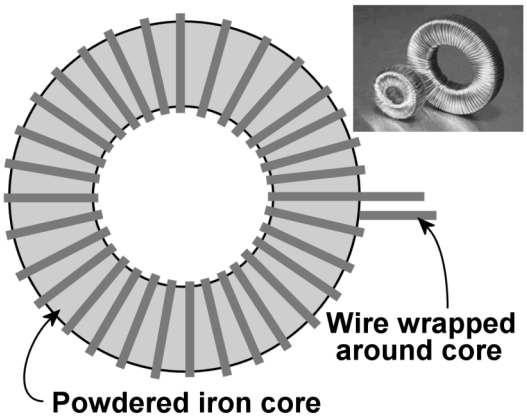


Fig. A5-4

The choke is simply a hollow bead (a torus) of powdered iron, and the power supply wire is wound as many times as you can around its circumference. The alternating power supply voltage has a frequency of between 50 and 60 cycles each second, and small inductance of the choke will have little or no effect at this low frequency. However, EMI harmonics have high frequencies, and it is these that we want to eliminate. At these frequencies, the electromagnetic coupling between the wire and the choke is significant, and generates eddy currents in the powdered iron. This dissipates the energy of these harmonics as heat in the iron. It is for this reason that powdered iron is to be used for this purpose. Ferrite cores are often used in radio and television circuits, but that is because such material is better "matched" to deal with extremely high frequencies. The harmonics generated by power switching transients are better dealt with by the relatively coarser structure of powdered iron.

Components

2 x BTA140-600 triacs (115/120V application)
2 x BT130-600 triacs (240V application)
2 x MOC3041 optoisolator zero crossing triac triggers
1 x 74HC00N CMOS 4x2input NAND gate
2 x 100 microfarad 63VDC electrolytic vapacitors (radial.end connectors)
2 x 4.7 nanofarad (0.0047 microfarad) high voltage disc ceramic capacitors
4 x 330 ohm 1/4 watt carbon resistors
2 x 180 ohm 1/4 watt carbon resistors
2 x 5.6 kilohm 1/4 watt carbon resistors
1 x 50 kilohm single gang linear potentiometer
1 x matching knob for potentiometer (preferably with calibration marks)
1 x 3mm red LED
1 x 3mm green LED
2 x iron powder toroid (14.8mm od x 8mm id x 6,35mm ht, or thereabouts)
1 x fuseholder
1 x fast-blow fuse (plus spares) 10 amp or 10 amp - see text
1 x stripboard (size to suit) predrilled 2.54mm hole spacing (eg Veroboard® type)
2 x power supply surface sockets
1 x 3-wire interconnecting lead
2 x panel mounted sockets to match interconnecting lead
2 x 8 pin DIL sockets

Figure A5-05 Example of a completed controller

A photograph of an actual controller, made by a skilful friend of one of the authors, may give you some idea of what one can look like. Note that the black control box is completely isolated from all power supply voltages.

A final word

We have deliberately made the layout of the circuits large. This has been done for two reasons:

- It makes construction easy
- It minimizes the risk of short circuits

Many amateur electronics enthusiasts vie with each other to produce the smallest layout they can. This goes back to the Good Old Days of discrete components, before integrated circuits were even dreamed of. To cram a one or two transistor radio into a match box was considered the height of skill, and sometimes the most amazing techniques were used to achieve this goal. However, more faults in electronic circuits can be traced back to cramming components too close together than any other single reason. There is absolutely no sense in attempting such miniaturization when making this controller. The only time we will be concerned about placing one component close to another will be when wiring in the choke, and that simply because the closer we can get it to the possible source of EMI the better.

Make the control module first, and check that it works as it should before making the power module. The control module is a totally independent circuit and will work quite happily on its own even when the signals it generates are not used to control another circuit. In contrast, the power module will just sit there and do nothing is it's not fed with a reliable set of signals from the control module, so you can't test it until you know that the control module is working properly. If nothing else, the skills you practice in making the control module first will serve you well when you reach the stage of making the power module and face the job of soldering those power triacs in place!

APPENDIX 6
VAPOR MANAGEMENT

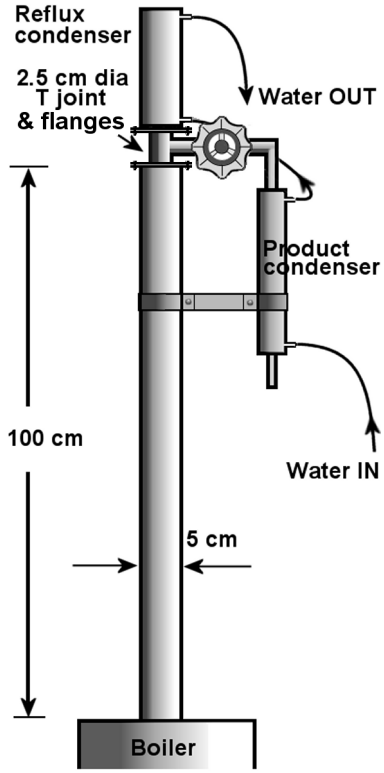


Fig. A6-1

A vapor management still head is very easy to build because the concept is so simple, and is a considerable advance on all previous liquid management techniques that utilize small, temperamental needle valves that can be difficult to procure and which often change their settings with temperature.

It has three further advantages:

- once calibrated its settings remain the same
- it maintains a constant selected reflux ratio for any boiler power setting or vapor composition
- the quantity of product distillate automatically reduces at the end of a run when the tails begin to be evident.

Equipment required

You need a boiler (with heater element control), a packed fractionating column, and two condensers: a reflux condenser for the top of the column, and a small Liebig condenser for collecting product. You also need a way of hooking them all together and managing the vapor flow. This is done by using a vapor control head.

You can make the head by placing a 2.5 cm (1") diameter tee between the top of the column and the reflux condenser. This is a standard plumbing part, and should be available at any hardware store. In the picture, the ends of the tee are joined to flanges, but you could easily use 5x2.5cm (2"x1") reducing couplings or end caps with holes for the tee drilled in them. (Use your imagination!)

NOTE: the vapor control head must not be significantly cooled by its proximity to the top reflux condenser, for if any of vapor condenses inside it then the separation principle will be compromised. For this reason, the best types of condenser to use are often a simple reflux coil or a cold finger as the enclosing shroud or sleeve, which is usually soldered or otherwise fastened to the separator, is not actively cooled. A poor choice would be a gloved cold finger, as the active cooling of the outer sleeve could easily extend to the separator.

The gate valve

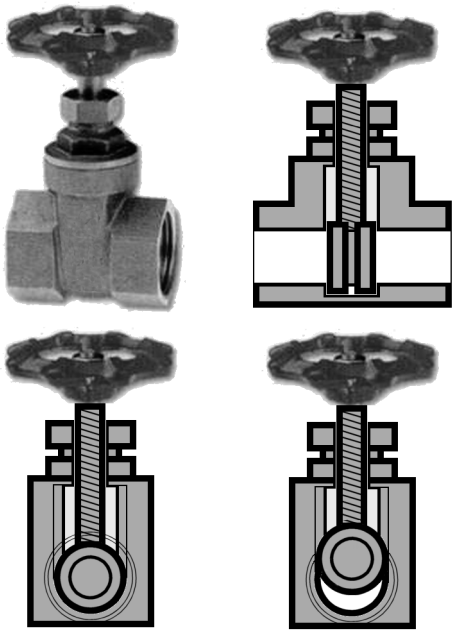


Figure A6-2

A 2.5cm (1”) **gate valve** controls the size of the opening through which vapor flows to the product condenser. This valve is at the heart of vapor management, and allows adjustment of the reflux ratio**.** As its name implies, it works by sliding a flat plate (or 'gate') across the whole width of the tube that runs through it. Gate valves (sometimes called 'slide' valves) are readily available from any plumbing or hardware store as they are a commonly used item. It is connected to the side arm of this junction, and then to an elbow to the other side of the valve. A small Liebig condenser is attached to this elbow. This condenser doesn't have to be big, as it will usually only have to cope about half the heat output of the heater when at its "production" setting of around 750 to 1000 watts. If you want to use the still as a stripper as well, then the rating you should aim for is around half the heat output of the heater when running on full power. A 1 cm (3/8 inch) diameter central tube running through about 45 cm (18 inches) of water jacket pipe should be adequate for all applications.

Attach the Liebig condenser the column of the still with a strut to avoid putting undue strain on the top joints.

Operation

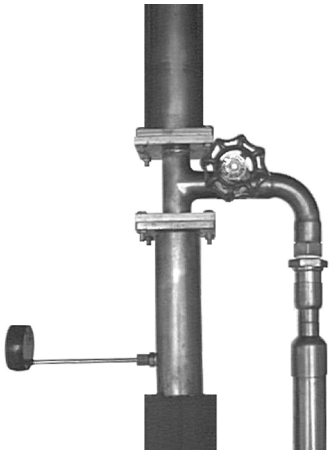


Fig. A6-3

That's it! Operation is equally simple. Close the gate valve and follow exactly the same procedure as for any other type of compound still (Chapter 5). When the time comes to draw off the heads, open the gate valve slowly until you are collecting distillate at the rate we recommend at this stage, which is about 1 or 2 drops per second. You may notice the only drawback of this system at this point – there is a lag of around 10 seconds after adjusting the valve before you see any change in the amount of distillate coming out of the product condenser. You will soon get used to this and find the still very easy to adjust. The gate valve allows you to make fine adjustments in the reflux ratio with coarse adjustments of the valve. One full turn of the valve wheel represents only about 2 or 3% change to the reflux ratio, and delicate control is much easier to achieve than with the small needle valve used with the liquid sampling method.

In fact, because the reflux ratio is not affected by vapor composition or production rate, you can easily calibrate the still by observing how your still operates at a known power with water. For each turn of the valve handle, collect product for 10 minutes and measure the quantity. You can calculate the % reflux for each valve setting by comparing the collected distillate to the amount of water being vaporized in the boiler. Because this design directly partitions the vapor, the reflux ratio at a particular setting will remain constant regardless of boiler power or the composition of the liquid in the boiler

Once the heads have been removed, then you can open up the gate valve even more and collect at whatever rate you choose. Because the two vapor paths and the valve are all the same size (1”), the minimum reflux ratio you can achieve is 50%. You will have full and complete control from 50% through 100% reflux.

At the end of the run, you will notice the temperature at the top of the column rises and the rate of collection drops off. This is because the volume of the distillate decreases as its percentage of water rises.
This gives you an independent test for the end of the main run, but slows down the collection of tails for future reprocessing. Fortunately, you can simply open the gate valve all the way and collect the tails quickly.

The vapor management head also works as an excellent stripper if you operate it with the gate valve fully open and collect raw material at a reflux ratio of around 50% with the boiler on full power. Due to the reflux action, the stripping run will produce a higher percentage product than a simple pot still, and this will save time later during the purification run.

APPENDIX 7

SCALING UP AND SPEEDING UP

Throughout this book, we have repeatedly offered guidelines for sizing and operating small-scale distilling apparatus. These guidelines (3.75 – 5 cm column diameter, 750-1000 Watts operating power once at temperature) are admittedly conservative, but are known to be safe, reliable and to produce an excellent quality product with minimal attention and intervention. These guidelines are an excellent starting point if you're interested in producing high-quality ethanol for botanical extracts, beverages or perfumes.

Many people are interested in distilling ethanol for fuel use, an activity that is even legal in the United States (but under strict licensing requirements). Fuel alcohol must be high purity to burn correctly, but must also be made in large quantities. Most fuel alcohol stills operate continuously, and have columns 150 mm (six inches) in diameter.

We recognize that in any activity or hobby, some people are primarily interested in the process: how to perfect it, how to expand it, how to best run it and how to speed it up. This interest and spirit of enquiry leads to new discoveries including better methods and equipment for us all. For those of you interested in scaling up or speeding up the process, we offer two words of caution, and then some helpful information.

Before you build a bigger mousetrap, consider carefully what you really need and want! This sounds simple and straightforward, but more than a few home brewers have built equipment that turned out to be much bigger than any batch they really wanted to make. Size your equipment to produce the actual amount of products you will use. Yes, you can build a 100 liter (25 gallon) boiler with a 150mm (six inch) column, and produce 20 liters (5 gallons) of 95% ethanol in eight hours, but what are you going to do with it? Fuel a car? Go for it (with your fuel producer's license securely in your pocket)! Store it for years until it is used up? Probably not a great idea.

As you scale up any process, interesting things happen. Within certain boundaries, you can increase all the parameters proportionately, and things will work well. At a certain size, though, things will suddenly quit working properly, and a complete redesign is needed. You have entered a new **operational domain**, where the ground rules are different. Once you have a design that works well in that domain, it can be scaled up easily until you find the upper edge of the domain.

Scaling up

For small scale distilling, the primary factors that can be easily altered are boiler power and column diameter. Column length will depend on the efficiency of separation, but if the packing is not altered too much then it doesn't make much difference, and 100-120cm (3 to 4 feet) is fine. Change the type of packing, and you're in a whole new ball game.

As column diameter increases, the capacity of the column and the area you are trying to distribute reflux over both increase dramatically (by the square of the diameter). We know of many people running columns in the 75-150mm (3-6 inch) range, and many of the people using 100mm (4 inch) and larger have had to resort to trays or other liquid spreading devices to get adequate performance. Scrubbers can be used to pack a 75mm column, but it takes a lot of them, and they have to be pulled out to a pancake shape with great care. A random filling like Raschig rings, or similar commercial packing material, is easier to use with large columns (please don't use marbles – see Chapter 8)

A second issue with increased column diameter is that the quality of separation can suffer, because turbulence within the column has more room to express itself and cause mixing between the theoretical

plates. This is one of the reasons that large industrial columns use physical plates to hold liquid in place and force vapor into contact with it under strictly controlled conditions.

As boiler power is increased, more vapor and liquid condensate are produced (45 liters of vapor every minute for each kilowatt of power). If column diameter is increased proportionately, the main problem will be assuring the even spread of the condensate across the width of the column. If column diameter is not increased, two phenomena can appear when the power becomes too high. These are **channeling** and **choking**.

Channeling occurs when the downward flow of reflux becomes large enough that it doesn't spread evenly over the packing material, but begins to flow downward as a rapid stream. This is because the layer of liquid spread over the packing material becomes too thick for wetting forces to hold it to the packing, and it begins flowing rapidly down the column. Once channeling has started, it will continue even though the boiler power or reflux rate is turned down dramatically, because a **preferred path** through the packing has been established. Channeling dramatically reduces the efficiency of the column and the purity of the product. About the only way to detect channeling is through the temperature of the vapor at the top of the column and the quality of the product. If you are using scrubbers, the flow level that will trigger channeling changes every time you re-pack the column, so the best advice is to operate well below the maximum possible power.

Choking is caused when the volume of liquid working its way down the column fills enough of the column area that vapor cannot flow freely upward. Choking is hard to miss, because the column begins to literally rumble and shake, and large puffs of vapor can shoot out of the condenser. Choking not only ruins the operation of the still, it can be **dangerous**! Channeling does **not** always occur before choking – one is a surface area issue, and the other is an open area issue.

Speeding up

One way of speeding up the operation of an existing still is to increase the boiler power. As long as you do not get into channeling or choking, this will work very well. Many people have reported being able to run a 50mm (2") column at 2 kW, and a 75mm (3") one at 4 kW. We would not recommend trying power levels any higher than these!

Your condenser(s) must be able to handle the vapor load created at higher power. The standard reflux coil can easily handle 2000 to 3000 Watts, depending upon cooling water temperature and flow rate. You should test this by distilling some **water** to make sure yours is adequate (see Chapters 3 and 4 where we discuss matching boiler and condenser performance).

If you are using electric heating and know the power being applied, you can calculate the new rate of vapor production and hence the amount of product to capture to maintain the desired reflux ratio. If you don't know the exact power you are using, you will need to determine the new rate of production by temporarily reducing the reflux to zero (best done with just water in the still) and collecting 100% of the condensate. When you know how many ml/min of condensate are being produced, you will know how to set the still to achieve the desired ratio. However, if using liquid management, please remember that the reflux ratio will vary with the composition of the vapor because you are drawing off liquid at a constant rate. If you draw off 4 ml of water every minute when testing, then you need to compensate for the lower density of ethanol, and draw off $4 \div 0.8 = 5$ ml of 96% ethanol every minute to maintain the same reflux ratio.

If you are using a vapor management still head then matters are simplified, because the valve settings are much simpler than with a needle valve and, once set, the reflux ratio will not vary. You can calibrate your valve once, and your settings will be good under all operating conditions.

The closer you operate to the edge of channeling or choking, the more watchful you will have to be in operating your still. We have talked about the tradeoff between time and quality – this tradeoff is between time and effort.

Once you are familiar and comfortable with the maximum operating power for your column, then you can increase the speed of a run by reducing the reflux ratio. You must do this very carefully, because

the reflux ratio can have a direct impact on quality. It is possible in some stills to produce 95% ethanol with reflux ratios as low as 35-40%, but it will not necessarily be as "clean" as ethanol produced at 90% reflux. The only way to know the capabilities of your still is to experiment.

The best way to do this experiment is to establish a run at 90% reflux and collect some product. Then switch collecting bottles and reduce the reflux ratio to 80%. Allow the still to operate for ten or fifteen minutes to stabilize under the new conditions, switch bottles again, and collect some 80% reflux product. Repeat the process for 70%, 60%, etc. It is important to let the still stabilize at each reflux ratio before collecting sample that you will test. Watch the temperature of the vapor like a hawk! As soon as it starts to rise, you are no longer making 95% ethanol, and have entered the regime of a fractionating still instead of a compound still.

You can continue the experiments in the fractionating regime, but you will now be making product more suitable for aging into a whiskey or brandy than pure ethanol.

After collecting all the samples, compare the samples taken under stable conditions at each reflux ratio for taste and smell. If you can tell no difference at all, you might be able to safely operate at lower than 90% reflux. Every still and every operator are different, and only you can make this decision.

Before you make a firm commitment to a lower reflux ratio, the final test is to invite friends to carry out an independent test to see if their palate can detect any differences between the samples. If you do lower your reflux ratios, the final test is whether the product produces a headache the next day. Congeners are the primary source of hangovers, and removing them completely is the main purpose of the compound still.

A Worry-Free Speedup

Once again, we introduce a trade-off, this time between time and money. A safe and worry free way to significantly speed up the process of compound distillation, without stressing the column or reducing the reflux ratio, is to **add columns**. Using our guidelines of 1 kW of power for a 50mm (2") column, you could run with a 3 kW element continuously at full power if you mounted three 50mm columns to the still, each with their own condenser. A single condenser could be used, but only if you devise a way of splitting the reflux into three equal streams to feed back to the columns - a design challenge for you! The heat-up time would be the same, the equilibration time would be the same, but the main run would only take 1/3 as long while producing the best possible product.

APPENDIX 8

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The Authors

Michael Nixon

Mike is a Chartered Engineer, a Member of the Institution of Electrical Engineers, and proud to be a Kiwi. His grounding was in physics, chemistry and mathematics, leading to a career as an engineering officer in the Royal Air Force in England. He is now retired and living in Auckland, back home in New Zealand. Like many Kiwi Blokes, his pride and joy is his Shed. A Shed is a place where a Kiwi Bloke spends much of his time alone surrounded by his tools, current and past half finished projects and the collection of parts and material usually referred to by others (typically wives) as 'that pile of junk'. It is a place where new uses for No. 8 fencing wire can be explored - for New Zealand is a place where lateral thinking and a healthy disregard for 'experts' is admired. For a peek into his Shed, see the picture at the beginning of Chapter 7.

Mike is the one who's first boiler was made from an old industrial vacuum cleaner, and who investigated the possibilities of using jam jars to make a glass column. In case you're wondering … industrial vacuum cleaners make great boilers, and jam jars can be made to work, but require tapered lapped joints to be ground using a lathe and lots of patience, using techniques borrowed from amateur astronomers who grind their own mirrors. This Shed 'research' led to development of vapor management techniques for compound distillation, to a new and simpler design of Soxhlet extractor, and is now progressing, in close collaboration with Mike McCaw, towards development of new and innovative types of still and processing equipment.

Michael McCaw

Mike McCaw is a research scientist for a major manufacturing company, specializing in the optimization of processes. He grew up on a dairy farm, where he learned how to improvise with materials at hand to keep a process running. 400 angry cows waiting to be milked can definitely focus one's attention! His undergraduate work was in botany, and his graduate work in Mycology, which led to his owning and operating a mushroom farm at one time.

He has been making beer, wine, mead, and a variety of other fermented beverages and food products for over twenty years, and maintains a large collection of yeast cultures. He, too, is an inveterate tinkerer with a workshop full of projects under way.

His profession consists of finding ways to make large machines and systems do more with less – to simplify, improve and stabilize them. He has eagerly applied these skills to beverage making, botanicals extraction and purification, and recently to the design of distillation equipment. Frustrated with the provincialism of many hobby groups, He and Mike Nixon have founded the Amphora Society, dedicated to widespread experimentation and sharing of knowledge in the areas of beverages, perfumes, extracts and foods.